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TITLE: LIQUID CRYSTAL COMPOUND WITH NEGATIVE PERMITIVITY ANISOTROPY VALUE, LIQUID CRYSTAL COMPOSITION CONTAINING THE LIQUID CRYSTAL COMPOUND, AND LIQUID CRYSTAL DISPLAY ELEMENT USING THE LIQUID CRYSTAL COMPO

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#### ABSTRACT:

PROBLEM TO BE SOLVED: To obtain the subject new compound capable of being applied to preparation of a liquid crystal composition for various kinds of display systems, including a vertical orientation system, etc., using a compound or composition having a permittivity anisotropy value being not only negative but also positive.

SOLUTION: This compound is a liquid crystal one of formula I [R1 and Y are an 1-30C straight chain or branched chain alkyl or the like.; rings A1 to A5 are separately a silacyclohexane ring, a ring of formula II (W1 and W2 are separately F or C1) or the like; X1 to X4 are separately a single bond, an 1-4C alkylene; R2 and R3 are each H, halogen or the like; a1 to a5 are separately 0 or 1], e.g. a compound of formula III. A compound of formula I is obtained e.g. through the processes of a formation of the ring of formula II, formation of a silacyclohexane ring, introduction of a difluoromethyleneoxy, etc. According to this, there provides a liquid crystal compound having a low viscosity and negative permittivity anisotropy value whose absolute value is large, having a controlled optical anisotropy value and high specific resistance, etc., and stable against heat and ultraviolet irradiation.

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#### (54) LIQUID-CRYSTALLINE MEDIUM

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#### **Publication Classification**

#### (7) ABSTRACT

The invention relates to a liquid-crystalline medium based on a mixture of polar compounds of negative dielectric anisotropy which comprises at least one compound of the formula I1

$$R^{11}$$
 $H$ 
 $Z$ 
 $F$ 
 $R^{12}$ 

and at least one compound of the formula I2

in which  $R^{11}$ ,  $R^{12}$ ,  $R^{21}$  and Z are as defined in claim 1, and the use thereof for an active matrix display based on the ECB or IPS effect.

[1

#### LIQUID-CRYSTALLINE MEDIUM

[0001] The invention relates to a liquid-crystalline medium based on a mixture of polar compounds having negative dielectric anisotropy, which comprises at least one compound of the formula 11

$$\mathbb{R}^{11} \underbrace{\qquad \qquad }_{\mathbb{R}^{12}} \mathbb{R}^{12}$$

[0002] and at least one compound of the formula I2

[0003] in which

[0004] R<sup>11</sup>, R<sup>12</sup> and R<sup>21</sup> are each, independently of one another, an alkyl or alkenyl radical having up to 15 carbon atoms which is unsubstituted, monosubstituted by CN or CF<sub>3</sub> or at least monosubstituted by halogen, where one or more CH<sub>2</sub> groups in these radicals may also, in each case independently of one another, be replaced by —O—, —S—,

[0005] —C=C—, —CO—, —CO—O—, —O—CO— or —O—CO—O— in such a way that 0 atoms are not linked directly to one another,

[0006] Z is 
$$-C_2H_4-$$
,  $-CH=CH-$ ,  $-CF_2O-$ ,  $-OCF_2-$  or a single bond, and

[0007] alkenyl is a straight-chain alkenyl radical having 2-6 carbon atoms.

[0008] Such media are particularly suitable for electrooptical displays with active matrix addressing based on the ECB effect, and for IPS (in-plane switching) displays.

[0009] The principle of electrically controlled birefringence, the ECB effect or alternatively DAP effect (deformation of aligned phases), was described for the first time in 1971 (M. F. Schieckel and K. Fahrenschon, "Deformation of nematic liquid crystals with vertical orientation in electrical fields", Appl. Phys. Lett. 19 (1971), 3912). This was followed by papers by J. F. Kahn (Appl. Phys. Lett. 20 (1972), 1193) and G. Labrunie and J. Robert (J. Appl. Phys. 44 (1973), 4869).

[0010] The papers by J. Robert and F. Clerc (SID 80 Digest Techn. Papers (1980), 30), J. Duchene (Displays 7

(1986), 3) and H. Schad (SID 82 Digest Techn. Papers (1982), 244) have shown that liquid-crystalline phases must have high values for the ratio between the elastic constants  $K_3/K_1$ , high values for the optical anisotropy  $\Delta n$  and values for the dielectric anisotropy  $\Delta e$  of from -0.5 to -5 in order to be suitable for high-information display elements based on the ECB effect. Electro-optical display elements based on the ECB effect have a homeotropic edge alignment. Dielectrically negative liquid-crystal media can also be used in displays utilizing the so-called IPS effect.

[0011] Technical use of this effect in electro-optical display elements requires LC phases which must satisfy a multiplicity of requirements. Particularly important here are chemical resistance to moisture, air and physical effects, such as heat, radiation in the infrared, visible and ultraviolet regions and direct and alternating electric fields.

[0012] Technically suitable LC phases are furthermore required to have a liquid-crystalline mesophase in a suitable temperature range and low viscosity.

[0013] None of the series of compounds having a liquid-crystalline mesophase which have been disclosed hitherto includes a single compound which meets all these requirements, In general, therefore, mixtures of from 2 to 25, preferably from 3 to 18, compounds are prepared in order to obtain substances which can be used as LC phases. However, optimum phases could not be prepared easily in this way, since no liquid-crystalline materials of significantly negative dielectric anisotropy were hitherto available.

[0014] Matrix liquid-crystal displays are known. Nonlinear elements which can be used for individual switching of the individual pixels are, for example, active elements (i.e. transistors). This is then referred to as an "active matrix", and a distinction can be made between two types:

[0015] 1. MOS (metal oxide semiconductor) transistors on a silicon wafer as substrate.

[0016] 2. Thin-film transistors (TFTs) on a glass plate as substrate.

[0017] In the case of type 1, the electro-optical effect used is usually dynamic scattering or the guest-host effect. The use of single-crystal silicon as the substrate material limits the display size, since even modular assembly of various part-displays results in problems at the joints.

[0018] In the case of more promising type 2, which is prefer-red, the electro-optical effect used is usually the TN effect.

[0019] A distinction is made between two technologies: TFTs comprising compound semiconductors, for example CdSe, or TFTs based on polycrystalline or amorphous silicon. Intensive work is being carried out worldwide on the latter technology.

[0020] The TFT matrix is applied to the inside of one glass plate of the display, while the other glass plate carries the transparent counterelectrode on its inside. Compared with the size of the pixel electrode, the TFT is very small and has virtually no adverse effect on the image. This technology can also be expanded to fully colour-compatible displays, in which a mosaic of red, green and blue filters is arranged in such a way that each filter element is located opposite a switchable pixel.

[0021] The TFT displays usually operate as TN cells with crossed polarizers in transmission and are backlit.

[0022] The term MLC displays here covers any matrix display containing integrated non-linear elements, i.e., besides the active matrix, also displays containing passive elements, such as varistors or diodes (MIM=metal-insulator-metal).

[0023] MLC displays of this type are particularly suitable for TV applications (for example pocket TVs) or for highinformation displays in automobile or aircraft construction. Besides problems regarding the angle dependence of the contrast and the response times, difficulties also arise in MLC displays due to inadequate resistivity of the liquidcrystal mixtures [TOGASHI, S., SEKIGUCHI, K., TANABE, H., YAMAMOTO, E., SORIMACHI, K., TAJIMA, E., WATANABE, H., SHIMIZU, H., Proc. Eurodisplay 84, Sept. 1984: A 210-288 Matrix LCD Controlled by Double Stage Diode Rings, p. 141 ff, Paris; STROMER, M., Proc. Eurodisplay 84, September 1984: Design of Thin Film Transistors for Matrix Addressing of Television Liquid Crystal Displays, p. 145 ff, Paris]. With decreasing resistance, the contrast of an MLC display drops. Since the resistivity of the liquid-crystal mixture generally drops over the life of an MLC display owing to interaction with the interior surfaces of the display, a high (initial) resistance is very important for displays which must have acceptable resistance values over a long service life.

[0024] The disadvantage of the MLC-TN displays disclosed hitherto is due to their comparatively low contrast, relatively high viewing-angle dependence and the difficulty of generating grey shades in these displays.

[0025] EP 0 474 062 discloses MLC displays based on the ECB effect. However, the LC mixtures described therein, which are based on 2,3-difluorophenyl derivatives containing an ester, ether or ethyl bridge, have low "voltage holding ratio" (HR) values after UV exposure.

[0026] There thus continues to be a great demand for MLC displays which have very high resistivity at the same time as a broad operating temperature range, short response times and a low threshold voltage which can be used to produce various grey shades.

[0027] It is an object of the invention to provide MLC displays based on the ECB effect which do not have the abovementioned disadvantages, or only do so to a lesser extent, and at the same time have very high resistivities.

[0028] It has now been found that this object can be achieved if nematic liquid-crystal mixtures comprising at least one compound of the formula I1 and one compound of the formula I2 are used in these display elements.

[0029] The invention thus relates to a liquid-crystalline medium based on a mixture of polar compounds having negative dielectric anisotropy which comprises at least one compound of the formula I1 and at least one compound of the formula I2.

[0030] The mixture according to the invention has very favorable values for the capacitive threshold, relatively high values for the holding ratio and at the same time very good low-temperature stability.

[0031] Some preferred embodiments are mentioned below:

[0032] a) A medium which additionally comprises one or more compounds of the formula II:

 $\mathbb{R}^2 - \left\{ \begin{array}{c} \\ \\ \\ \\ \\ \end{array} \right\}_p = \left\{ \begin{array}{c} \\ \\ \\ \\ \end{array} \right\} - \left( O \right) C_v H_{2v+1} \\ \\ \end{array}$ 

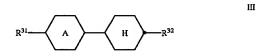
[0033] in which

[0034]  $R^2$  is independently as defined for  $R^{11}$ ,  $R^{12}$  and  $R^{21}$ ,

[0035] p is 1 or 2, and

[0036] v is from 1 to 6.

[0037] b) A medium which additionally comprises one or more compounds of the formula III:



[0038] in which

[0039]  $R^{31}$  and  $R^{32}$  are each, independently of one another, a straight-chain alkyl or alkyloxy radical having up to 12 carbon atoms, and

[0040] c) A medium which comprises two, three, four or more, preferably two, three or four, compounds of the formula 11.

[0041] d) A medium which comprises at least two compounds of the formula I2.

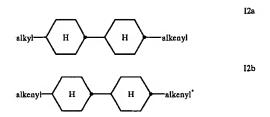
[0042] e) A medium in which the proportion of compounds of the formula I1 in the total mixture is at least 10% by weight, preferably at least 20% by weight.

[0043] f) A medium in which the proportion of compounds of the formula 12 in the total mixture is at least 5% by weight, preferably at least 10% by weight.

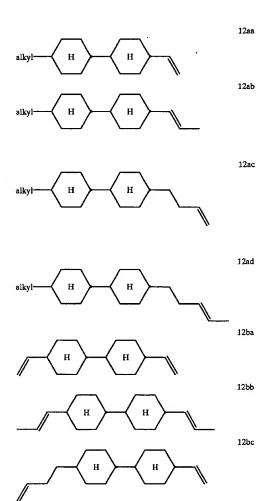
[0044] g) A medium in which the proportion of compounds of the formula II in the total mixture is at least 20% by weight.

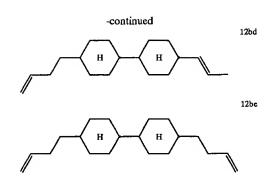
[0045] h) A medium in which the proportion of compounds of the formula III in the total mixture is at least 5% by weight.

[0046] i) A medium which comprises at least one compound selected from the formulae I2a and I2b.



[0047] Particular preference is given to the compounds of the formulae I2aa-I2ad and I2ba-I2be:





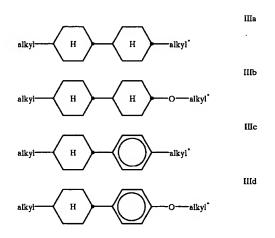
[0048] in which

[0049] alkenyl and

[0050] alkenyl\* are each, independently of one another, a straight-chain alkenyl radical having 2-6 carbon atoms, and

[0051] alkyl is a straight-chain alkyl radical having 1-6 carbon atoms.

[0052] j) A medium which additionally comprises a compound selected from the formulae IIIa to IIId:



[0053] in which

[0054] alkyl and

[0055] alkyl\* are each, independently of one another, a straight-chain alkyl radical having 1-6 carbon atoms.

[0056] The medium according to the invention preferably comprises at least one compound of the formula IIIa and/or formula IIIb.

[0057] k) A medium which essentially consists of: 10-40% by weight of one or more compounds of the formula II,

[0058] 5-30% by weight of one or more compounds of the formula 12, and

[0059] 20-70% by weight of one or more compounds of the formula II.

[0060] 1) A medium which additionally comprises one more compounds of the formulae

$$R^2$$
 $H$ 
 $F$ 
 $F$ 
 $F$ 
 $F$ 
 $F$ 
 $F$ 
 $H$ 
 $C_xH_{2x+1}$ 

[0061] in which

[0062] R<sup>7</sup> and R<sup>8</sup> are each, independently of one another, as defined for R<sup>11</sup> R<sup>12</sup> and R<sup>21</sup> in claim 1, and

[0063] w and x are each, independently of one another, from 1 to 6.

[0064] m) A medium which additionally comprises one more compounds of the formulae

$$R^{13} \longrightarrow H \longrightarrow F \longrightarrow (CH_2)_z - O - C_mH_2$$

$$R^{14} \longrightarrow H \longrightarrow F \longrightarrow (CH_2)_z - O - C_mH_2$$

$$R^{15} \longrightarrow H \longrightarrow F \longrightarrow F$$

$$R^{16} \longrightarrow H \longrightarrow CF_2O \longrightarrow (O) \longrightarrow alkyl$$

[0065] in which  $R^{13}$ - $R^{22}$  are each, independently of one another, as defined for  $R^{11}$ ,  $R^{12}$  and  $R^{21}$ , and z and m are each, independently of one another, 1-6.  $R^E$  is H, CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub> or n-C<sub>3</sub>H<sub>7</sub>.

[0066] n) A medium in which the compound of the formula I1 is selected from the group consisting of I1a to I1g:

-continued

$$R^{11} \qquad H \qquad \qquad F \qquad F$$

$$R^{11} \qquad H \qquad \qquad F \qquad F$$

$$R^{11} \qquad H \qquad \qquad GCH = CH_2$$

$$R^{11} \qquad H \qquad GCH = CH_2$$

[0067] in which R<sup>11</sup> is as defined above, and s is 1-12. R<sup>11</sup> is preferably straight-chain alkyl having 1 to 6 carbon atoms, vinyl, 1E-alkenyl or 3E-alkenyl.

[0068] o) A medium which comprises one or more compounds of the formula I1a and/or I1g.

[0069] p) A medium which additionally comprises one or more compounds of the formula

[0070] in which R is alkyl, alkenyl, alkoxy or alkenyloxy each having 1 or 2 to 6 carbon atoms.

[0071] The invention furthermore relates to an electrooptical display having active matrix addressing based on the ECB effect, characterized in that it comprises, as dielectric, a liquid-crystalline medium as defined above.

[0072] The liquid-crystal mixture preferably has a nematic phase range of at least 60 K and a maximum flow viscosity  $V_{20}$  of 30 mm<sup>2</sup>.s<sup>-1</sup> at 20° C.

[0073] The liquid-crystal mixture according to the invention preferably has a  $\Delta\epsilon$  of from about -0.5 to -6.0, in particular from about -3.0 to -4.5, where  $\Delta\epsilon$  is the dielectric anisotropy. The rotational viscosity  $\gamma_1$  is preferably <225 mPa.s, in particular <180 mPa.s.

[0074] The birefringence  $\Delta n$  in the liquid-crystal mixture is generally between 0.04 and 0.13, preferably between 0.06 and 0.11, and/or the dielectric constant  $\epsilon_{\parallel}$  of greater than or equal to 3, preferably from 3.2 to 8.5.

[0075] The dielectrics may also comprise further additives which are known to the person skilled in the art and are described in the literature.

[0076] For example, 0-15% of pleochroic dyes can be added, furthermore conductive salts, preferably ethyldim-

ethyl-dodecylammonium 4-hexoxybenzoate, tetrabutylammonium tetraphenylborate or complex salts of crown ethers (cf., for example, Haller et al., Mol. Cryst. Liq. Cryst., Volume 24, pages 249-258 (1973)) for improving the conductivity, or substances for modifying the dielectric anisotropy, the viscosity and/or the alignment of the nematic phases. Such substances are described, for example, in DE-A 22 09 127, 22 40 864, 23 21 632, 23 38 281, 24 50 088, 26 37 430 and 28 53 728.

[0077] The individual components of the formulae I1, I2, II and III in the liquid-crystal phases according to the invention are either known or their modes of preparation can easily be derived from the prior art by the person skilled in the relevant art, since they are based on standard methods which are described in the literature.

[0078] The nematic liquid-crystal mixtures in the displays according to the invention generally comprise two components A and B, which themselves consist of one or more individual compounds.

[0079] Component A has significantly negative dielectric anisotropy and gives the nematic phase a dielectric anisotropy of ≤-0.3. It preferably comprises compounds of the formulae I1 and II.

[0080] The proportion of component A is preferably between 45 and 100%, in particular between 60 and 100%.

[0081] For component A, one (or more) individual compound(s) having a  $\Delta \epsilon \leq -0.8$  are preferably selected. The smaller the proportion of component A in the total mixture, the more negative this value must be.

[0082] Component B has pronounced nematogeneity and a flow viscosity of not more than 30 mm<sup>2</sup>.s<sup>-1</sup>, preferably not more than 25 mm<sup>2</sup>.s<sup>-1</sup>, at 20° C. It preferably comprises compounds of the formulae I2 and III.

[0083] Particularly preferred individual compounds of component B are extremely low-viscosity nematic liquid crystals having a flow viscosity of not more than 18 m<sup>2</sup>.s<sup>-1</sup>, preferably not more than 12 mm<sup>2</sup>.s<sup>-1</sup>, at 20° C.

[0084] Component B has monotropic or enantiotropic nematogeneity, has no smectic phases and can prevent the occurrence of smectic phases in liquid-crystal mixtures down to very low temperatures. If, for example, a smectic liquid-crystal mixture is mixed with various materials of high nematogeneity, the degree of suppression of smectic phases that is achieved can be used to compare the nematogeneity of these materials.

[0085] Numerous suitable materials are known to the person skilled in the art from the literature. Particular preference is given to compounds of the formula III.

[0086] In addition, these liquid-crystal phases can also contain more than 18 components, preferably from 18 to 25 components.

[0087] The phases preferably contain from 4 to 15, in particular 5 to 12, compounds of the formulae I1, I2, II and optionally III.

[0088] Besides compounds of the formulae I1, I2, II and III, it is also possible for other constituents to be present, for

example in an amount of up to 45% of the total mixture, but preferably up to 35%, in particular up to 10%.

[0089] The other constituents are preferably selected from nematic or nematogenic substances, in particular known substances, from the classes consisting of the azoxy-benzenes, benzylideneanilines, biphenyls, terphenyls, phenyl or cyclohexyl benzoates, phenyl or cyclohexyl cyclohexanes carboxylates, phenylcyclohexanes, cyclohexyl-biphenyls, cyclohexylcyclohexanes, cyclohexyl-naphthalenes, 1, 4-biscyclohexylbiphenyls or cyclohexyl-pyrimidines, phenyl or cyclohexyldioxanes, optionally halogenated stilbenes, benzyl phenyl ethers, tolans and substituted cinnamic acids.

[0090] Important compounds which can be used as constituents of liquid-crystal mixtures of this type can be characterized by the formula IV

[0091] in which L and E are each a carbocyclic or heterocyclic ring system from the group consisting of 1,4-disubstituted benzene and cyclohexane rings, 4,4'-disubstituted biphenyl, phenylcyclohexane and cyclo-hexylcyclohexane systems, 2,5-disubstituted pyrimidine and 1,3-dioxane rings, 2,6-disubstituted naphthalene, di- and tetrahydronaphthalene, quinazoline and tetra-hydroquinazoline, G is

[0092] or a C—C single bond, Q is halogen, preferably chlorine, or —CN, and R<sup>9</sup> and R<sup>10</sup> are each alkyl, alkenyl, alkoxy, alkanoyloxy or alkoxycarbonyloxy having up to 18, preferably up to 8, carbon atoms, or one of these radicals is alternatively CN, NC, NO<sub>2</sub>, NCS, CF<sub>3</sub>, F, Cl or Br.

[0093] In most of these compounds, R<sup>9</sup> and R<sup>10</sup> are different from one another, one of these radicals usually being an alkyl or alkoxy group. However, other variants of the proposed substituents are also common. Many such substances or mixtures thereof are commercially available. All these substances can be prepared by methods which are known from the literature.

[0094] It will be appreciated by a person skilled in the art that the ECB mixture according to the invention may also comprise compounds in which, for example, H, N, O, Cl or F have been replaced by the corresponding isotopes.

[0095] The construction of the liquid-crystal displays according to the invention corresponds to the conventional geometry, as described, for example, in EP-A 0 240 379.

[0096] Without further elaboration, it is believed that one skilled in the art can, using the preceding description, utilize the present invention to its fullest extent. The following preferred specific embodiments are, therefore, to be construed as merely illustrative, and not limitative of the remainder of the disclosure in any way whatsoever.

[0097] In the foregoing and in the following examples, all temperatures are set forth uncorrected in degrees Celsius; and, unless otherwise indicated, all parts and percentages are by weight.

[0098] The entire disclosure of all applications, patents and publications, cited above, and of corresponding German application No. DE 100 18 899.0, filed Apr. 14, 2000, is hereby incorporated by reference.

[0099] Besides the compounds of the formulae I1 and I2, the liquid-crystal mixtures according to the invention preferably comprise one or more of the compounds mentioned below.

[0100] The following abbreviations are used:

PCH-nOmFF CCP-nOmFF CCP-nmFF D-nOmFF CBC-nmF C<sub>m</sub>H<sub>2m+1</sub> CBC-nm  $C_mH_{2m+1}$ CCP-V-m CCP-Vn-m  $C_{m}H_{2m+1}$ 

-continued CPYC-n-m CYYC-n-m CCYY-n-(O)m CCH-nOm OC<sub>m</sub>H<sub>2m+1</sub> CY-n-m CCH-nm CmH2m+1 CC-n-V CC-n-V1 CP-nOmFF CH-nm Н CmH2m+1 CEY-V-n

-continued

$$CVY \cdot V \cdot D$$
 $CVY \cdot V \cdot D$ 
 $CVY \cdot V \cdot D$ 
 $CVY \cdot V \cdot D$ 
 $CVY \cdot D \cdot D$ 
 $CVY \cdot D$ 

CPY-V-Om

-continued

$$C_nH_{2n+1}$$
  $H$   $H$   $(CH_2)_z$   $O$   $C_mH_{2m+1}$   $CPY-n-Om$ 

$$C_nH_{2n+1}$$
— $C_mH_{2m+1}$ 

$$\begin{array}{c|c} & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$$

$$C_{n}H_{2n+1} - \underbrace{ H } \\ C_{n}H_{2n+1} - \underbrace{ C_{m}H_{2m+1} } \\ C_{m}H_{2n-1}O)m$$

$$C_nH_{2n+1}$$
  $H$   $CF_2O$   $F$   $O$   $C_mH_{2m+1}$ 

$$C_nH_{2n+1}$$
  $OCF_2$   $O-C_mH_{2m+1}$ 

$$CCQY-n-(O)m$$
 
$$C_nH_{2n+1} - H - CF_2O - OO-C_mH_{2m+1}$$

$$CPQY\text{-}n\text{-}(O)m$$
 
$$C_nH_{2n+1} - H - CF_2O - (O)-C_mH_{2m+1}$$

-continued

CPQIY-n-(O)m

$$C_nH_{2n+1} - \underbrace{H} - OCF_2 - F \\ CCY-V-(O)m$$

$$\begin{array}{c|c} & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$$

[0102] The abbreviations furthermore have the following meanings:

[0103] V.threshold voltage, capacitive [V] at 20° C.

[0104]  $\;\Delta n$  optical anisotropy measured at 20° C. and 589 nm

[0105] Δε dielectric anisotropy at 20° C. and 1 kHz c.p. clearing point [° C.]

[0106] γ<sub>1</sub> rotational viscosity measured at 20° C. [mPa.s]

[0107] LTS low temperature stability

[0108] The display used to measure the threshold voltage has two plane-parallel outer plates at a separation of 5  $\mu$ m and electrode layers covered by lecithin alignment layers on the inside of the outer plates, which produce a homeotropic alignment of the liquid crystal molecules.

#### MIXTURE EXAMPLES

#### Example 1

#### [0109]

18.0%	S → N:	<-40° C.
19.0%	Clearing point [° C.]:	69.5
8.0%	Δn [589 nm, 20° C.]:	+0.1011
7.0%	Δε [1 kHz, 20° C.]:	-3.3
8.0%	4 (1 kHz. 20° C.):	3.6
18.0%	γ <sub>1</sub> [mPa · s, 20° C.]:	115
12.0%	V <sub>o</sub> [V]:	2.10
10.0%	LTS in cells: At -20° C., -30° C., -40° C.	nem. > 1 000 h
	19.0% 8.0% 7.0% 8.0% 18.0% 12.0%	19.0% Clearing point [° C.]: 8.0% Δn [589 nm, 20° C.]: 7.0% Δε [1 kHz, 20° C.]: 8.0% ξη 1 kHz, 20° C.]: 18.0% γ1 [mPa·s, 20° C.]: 12.0% V <sub>0</sub> [V]: 10.0% LTS in cells:

#### Example 2

#### [0110]

PCH-304FF	19.0%	S → N:	<-40° C.
PCH-504FF	20.0%	Clearing point [° C.]:	71.0
CCP-302FF	6.0%	Δn [589 nm, 20° C.]:	+0.1020
BCH-32	7.0%	Δε [1 kHz, 20° C.]:	-3.9
CCH-35	5.0%	4 (1 kHz. 20° C.):	3.7
CC-3-V1	8.0%	€ [1 kHz, 20° C.]: Y1 [mPa·s, 20° C.]:	142

		continued		-continued			
CC-5-V CPY-2-02 CPY-3-02	12.0% At -:	in cells: n 20° C., and -30° C.	.92 em. > 1 000 h	CCH-35 CC-5-V D-302-FF D-502FF CPY-2-02 CPY-3-02	5.0% 6.0% 8.0% 8.0% 14.0% 12.0%	γ <sub>1</sub> [mPa·s, 20° C.]: V <sub>o</sub> [V]:	139 1.96
F01117	ŀ	Example 3					
[0111]					E	Example 7	
		<del> </del>	<del> </del>	[0115]			
PCH-304FF PCH-502FF PCH-504FF	8.0%	S → N: Clearing point [° C.]: An [589 nm, 20° C.]:	<-30° C. 75.5 +0.1005				
CCP-302FF CC-3-V1 CC-5-V CCH-35 CPY-2-02 CPY-3-02 BCH-32	10.0% 8.0% 13.0% 5.0% 12.0% 4.0%	Aε [1 kHz, 20° C.]: <sup>ε</sup>    [1 kHz, 20° C.]:  γ <sub>1</sub> [mPa·s, 20° C.]:  V <sub>o</sub> [V]:	-4.2 3.7 149 1.95	PCH-304FF PCH-502FF PCH-504FF CC-5-V CC-3-V1 CCH-35 CPY-2-02 CPY-3-02	14.0% 7.0% 18.0% 8.0% 8.0% 5.0% 12.0%	$S \rightarrow N$ : Clearing point [° C.]: $\Delta n$ [589 nm, 20° C.]: $\Delta \epsilon$ [1 kHz, 20° C.]: $\epsilon$ <sub>  </sub> [1 kHz, 20° C.]: $\gamma$ <sub>  </sub> [mPa · s, 20° C.]: V <sub>0</sub> [V]:	<-30° C. 80.5 +0.1006 -4.9 3.8 186 1.89
	-			CCP-302FF CCPC-33	13.0% 3.0%		
0112]	£	Example 4					·
VII.2)					E	example 8	
DOM SO IT	0.00			[0116]			
PCH-304FF PCH-502FF PCH-504FF CCP-302FF CCP-31FF CC-3-V1 CCH-35 CPY-2-02 CPY-3-02	8.0% 18.0%	S → N: Clearing point [° C.]: ∆n [589 nm, 20° C.]: ∆∈ [1 kHz, 20° C.]: ¶  [1 kHz, 20° C.]: ¬  [mPa·s, 20° C.]: V <sub>o</sub> [V]:	c-30° C. 83.5 +0.1022 -4.9 3.8 189 1.93	PCH-304FF PCH-502FF PCH-504FF CCH-35 CC-3-V1 BCH-32 CPY-2-02 CPY-3-02 CCP-302FF	14.0% 10.0% 17.0% 5.0% 9.0% 6.0% 13.0% 12.0% 14.0%	S → N: Clearing point [° C.]: An [589 nm, 20° C.]: A∈ [1 kHz, 20° C.]: ¶1 kHz, 20° C.]: ¶1 [mPa·s, 20° C.]: V <sub>o</sub> [V]:	<-30° C 80.0 +0.1104 -5.1 3.8 202 1.83
	E	Example 5					
0113]		•			E	xample 9	
				[0117]			
PCH-304FF PCH-504FF	11.0% 16.0%	S → N: Clearing point [° C.]:	<-30° C. 83.5				
CC-5-V PCH-302 CCH-35 CC-3-V1 CPY-2-02 CPY-3-02 CCP-302FF CCP-V2-1	12.0% 6.0% 5.0% 8.0% 12.0% 11.0% 7.0%	Δn [589 nm, 20° C.]: Δε [1 kHz, 20° C.]:  [1 kHz, 20° C.]:  [1 kHz, 20° C.]:  [2 [mPa · s, 20° C.]:  V <sub>o</sub> [V]:	+0.1006 -3.7 3.5 150 2.23	PCH-304FF PCH-502FF PCH-504FF CCP-302FF CPY-2-02 CPY-3-02 CCP-V2-1 CC-3-V1 CCH-35 CC-5-V	14.0% 8.0% 15.0% 8.0% 9.0% 10.0% 5.0% 8.0% 5.0%	S → N: Clearing point [° C.]: $\Delta n$ [589 nm, 20° C.]: $\Delta \epsilon$ [1 kHz, 20° C.]: $\epsilon_{\parallel}$ [1 kHz, 20° C.]: $\gamma_{1}$ [mPa·s, 20° C.]: $V_{o}$ [V]:	<-30° C. 70.0 +0.0906 -3.7 3.6 119 2.03
	E	xample 6			-5.0,0		<u> </u>
0114]		-			Ex	cample 10	
				[0118]			
PCH-502FF PCH-504FF	8.0% 16.0%	S → N: Clearing point [° C.]:	<-30° C. 70.5	DON 304EE	19.00%	S N:	200 0
PCH-301 CCP-V2-1 CC-3-V1	9.0% 5.0% 9.0%	Δn [589 nm, 20° C.]: Δε [1 kHz, 20° C.]: ¶[1 kHz, 20° C.]:	+0.1007 -4.2 3.9	PCH-304FF PCH-502FF PCH-504FF	18.0% 10.0% 15.0%	S → N: Clearing point [° C.]: An [589 nm, 20° C.]:	< -30° C 80.5 +0.1192

						Example 14	
		-continued		[0122]			
CCP-302F	F 10.0%	δ δε [1 kHz, 20° C.]	-5.1	_ , ,			
BCH-32	8.0%	•	4.0				
CCP-V-1	10.0%	γ <sub>1</sub> [mPa·s, 20° C.]	: 225	PCH-304FF		S → N:	< -40° C.
PCH-302	3.0%	v <sub>o</sub> [v]:	1.83	PCH-504FF BCH-32		Clearing point [° C.]: An [589 nm, 20° C.]:	69.0 +0.0978
PGIGI-3-F	2.0%	,		CCP-V-1		Δε [1 kHz, 20° C.]:	-3.0
CPY-2-02	12.0%	,		CC-3-V1 CC-5-V	8.0% 20.0%	ϵ <sub> </sub> [1 kHz, 20° C.]: V <sub>o</sub> [V]:	3.6 2.17
CPY-3-02	12.0%	•		CPY-2-02	10.0%	γ <sub>1</sub> [mPa·s, 20° C.]:	108
	_			_ CPY-3-02		LTS in cells: at -20° C., -30° C., -40° C	nem. > 1 000 h
		Enamela 11			***		
504407		Example 11				Example 15	
[0119]						Example 15	
				[0123]			
PCH-304F	F 15.0%	s → N:	< −30° C.				
PCH-504F		. • • • • • • • • • • • • • • • • • • •		PCH-304FF	16.0%	S → N:	< -30° C.
CCH-35 CC-5-V	5.0% 12.0%		-	PCH-504FF CCP-302FF	18.0% 6.0%	Clearing point [° C.]: An [589 nm, 20° C.]:	73.5 +0.0883
CC-3-V1	10.0%		3.6	CPY-2-02	6.0%	Δε [1 kHz, 20° C.]:	-3.1
BCH-32	8.0%	** *	2.04	CPY-3-02 CCP-V2-1	11.0% 10.0%	ε <sub>μ</sub> [1 kHz, 20° C.]: V <sub>o</sub> [V]:	3.4 2.26
CPY-2-02 CPY-3-02	10.0% 7.0%		: 145	CC-3-V1	8.0%	γ <sub>1</sub> [mPa · s, 20° C.]:	113
CPY-V-02	10.0%			CCH-35 CC-5-V	5.0% 20.0%	LTS in cells: at -20° C. and -30° C.	nem. > 1 000 h
CPY-V-04	8.0%	· ··					
						Example 16	
		Example 12				Example 10	
[01:20]		-		[0124]			
[0120]							
				PCH-304FF	13.0	% Clearing point [° C.]	<b>]:</b> 70
PCH-304FF	10.0%	S → N:	< -30° C.	PCH-502FF PCH-504FF	8.0° 11.0°		}: +0.0986 −3.2
PCH-504FF CCH-35	16.0% 5.0%	Clearing point [° C.]: \[ \Delta \] [589 nm, 20° C.]:	80.0 +0.1021	CPY-3-02	10.0	% $\epsilon_{\parallel}$ [1 kHz, 20° C.]:	3.6
CC-5-V CC-3-V1	20.0% 10.0%	$\Delta \epsilon$ [1 kHz, 20° C.]: $\epsilon_0$ [1 kHz, 20° C.]:	-3.5 3.5	CPQIY-3-02 CPQIY-3-04	5.0° 5.0°		2.12 116
BCH-32	3.0%	գլլաև, 20 с.յ. V <sub>o</sub> [V]։	2.17	CPY-2-02	9.0	%	
CPY-2-02 CPY-3-02	10.0% 10.0%	γ <sub>1</sub> [mPa·s, 20° C.]: LTS in cells:	131 nem. > 1 000 h	BCH-32 CC-3-V1	8.0° 8.0°		
CPY-V-02	10.0%	at -20° C., -30° C.	nem. > 1 000 n	CCH-35	5.0	%	
CCP-302FF	6.0%			- CC-5-V	18.0	<b>%</b>	
		Example 13				Example 17	
[0121]				[0125]			
PCH-304FF	14.0%	S → N:	< -30° C.	PCH-304FF	16.09		
PCH-504FF	15.0%	Clearing point [° C.]:	84.0	PCH-502FF PCH-504FF	8.09 12.09		: +0.0954 -3.4
CCY-V-02 CPY-3-1	10.0% 9.0%	Δn [589 nm, 20° C.]: Δε [1 kHz, 20° C.]:	+0.1140 -4.8	CPY-3-02	8.09	$\epsilon_{\parallel}$ [1 kHz, 20° C.]:	3.6
CC-3-V1	10.0%	ε <sub>μ</sub> [1 kHz, 20° C.]:	3.8	CPQY-3-02 CPQY-5-02	5.09 5.09		2.08 122
CCH-35 CC-5-V	5.0% 7.0%	V <sub>o</sub> [V]: γ <sub>i</sub> [mPa·s, 20° C.]:	1.94 183	CPY-2-02	9.09	%	***
CPY-V-02	10.0%	LTS in cells:	nem. > 1 000 h	BCH-32 CC-3-V1	8.09 8.09		
CPY-2-02 CPY-3-02	10.0% 10.0%	at -20° C.		CCH-35	5.09	%	
				CC-5-V	16.09	%o	

12

11

ш

Example 18

[0126]

PCH-304 PCH-502 PCH-504 CPY-3-02 CQY-5-1 CQY-3-04 CPY-3-04 BCH-32 CC-3-V1 CCH-35 CC-5-V	FF 10.0% FF 14.0% 12.0% 5.0% 4 5.0%	Clearing point [° C.]: Δn [589 nm, 20° C.]: Δε [1 kHz, 20° C.]: ε <sub>1</sub> [1 kHz, 20° C.]: V <sub>o</sub> [V]: γ <sub>1</sub> [mPa·s, 20° C.]:	70.0 +0.1023 -3.3 3.6 2.14

Example 19

[0127]

	PCH-304FF PCH-502FF PCH-504FF	11.0% 9.0% 16.0%	Clearing point [° C.]: Δn [589 nm, 20° C.]: Δε [1 kHz, 20° C.]	69.5 +0.0952 -3.6	
	rcn-304rr	10.070		-3.0	
•	CPQIY-3-02	8.0%	ε <sub>1</sub> [1 kHz, 20° C.]:	3.6	
	CPY-2-04	10.0%	Ÿ₀[V]	2.08	
	CPY-3-02	11.0%	γ <sub>1</sub> [mPa · s, 20° C.]	120	
	CCPC-33	3.0%	-		
	CC-3-V1	8.0%			
	CCH-35	5.0%			
	CC-5-V	19.0%			

Example 20

[0128]

_				
•	PCH-304FF	13.0%	Clearing point [° C.]:	70.5
	PCH-502FF	8.0%	Δn [589 nm, 20° C.]:	+0.0900
_	PCH-504FF	16.0%	Δ∈ [1 kHz, 20° C.]:	-3.7
~	CCQY-3-02	8.0%	e] [1 kHz, 20° C.]:	3.6
	CPY-2-02	10.0%	ν <sub>α</sub> [ν]:	2.06
	CPY-3-02	10.0%	γ <sub>1</sub> [mPa.s, 20° C.]:	119
	CCP-V2-1	4.0%	,,,	
	CC-3-V1	8.0%		
	CCH-35	5.0%		
	CC-5-V	18.0%		

1. A liquid-crystalline medium based on a mixture of polar compounds having negative dielectric anisotropy, comprising at least one compound of formula I1

$$R^{11}$$
 $H$ 
 $Z$ 
 $R^{12}$ 

and at least one compound of formula 12

$$R^{2l} \underbrace{ \qquad \qquad }_{H} \underbrace{ \qquad \qquad }_{alkenyl}$$

in which

R<sup>11</sup> R<sup>12</sup> and R<sup>21</sup> are each, independently of one another, alkyl or alkenyl having up to 15 carbon atoms which is unsubstituted, monosubstituted by CN or CF<sub>3</sub> or at least monosubstituted by halogen, where one or more CH<sub>2</sub> groups in these radicals may also, in each case independently of one another, be replaced by —O—, —S—.

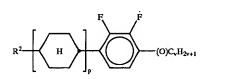
$$\overline{\phantom{a}}$$

—C=C—, —CO—, —CO—O—, —O—CO— or —O—CO—O— in such a way that O atoms are not linked directly to one another,

Z is 
$$-C_2H_4$$
,  $-CH=CH$ ,  $-CF_2O$ ,  $-OCF_2$  or a single bond, and

alkenyl is straight-chain alkenyl having 2-6 carbon atoms.

2. The medium according to claim 1, additionally comprising at least one compound of formula II



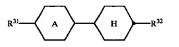
in which

R<sup>2</sup> is independently as defined for R<sup>11</sup>, R<sup>12</sup> and R<sup>21</sup>,

p is 1 or 2, and

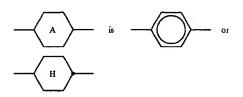
v is 1 to 6.

3. The medium according to claim 1, additionally comprising at lesst one compound of formula III



in which

R<sup>31</sup> and R<sup>32</sup> are each, independently of one another, a straight-chain alkyl or alkyloxy radical having 1-12 carbon atoms, and

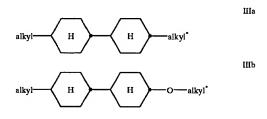


4. The medium according to claim 2, additionally comprising at lesat one compound of formula III

in which

R<sup>31</sup> and R<sup>32</sup> are each, independently of one another, a straight-chain alkyl or alkyloxy radical having 10 1-12 carbon atoms, and

- 5. The medium according to claim 1, comprising at least three compounds of formulae I1 or I2.
- 6. The medium according to claim 1, having a proportion of compounds of formula I1 in the total mixture of at least 10% by weight.
- 7. The medium according to claim 1, having a proportion of compounds of formula 12 in the total mixture of at least 5% by weight.
- 8. The medium according to claim 2, having a proportion of compounds of formula II in the total mixture of at least 20% by weight.
- 9. The medium according to claim 3, having a proportion of compounds of formula III in the total mixture of at least 5% by weight.
- 10. The liquid-crystalline medium according to claim 3, comprising at least one compound of formulae IIIa to IIId:



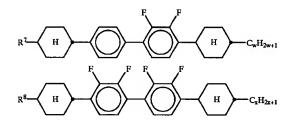
in which

alkyl and

alkyl\* are each, independently of one another, straightchain alkyl having 1-6 carbon atoms.

11. The liquid-crystalline medium according to claim 10, comprising at least one compound of formula IIIa, at least one compound of formula IIIb, or a mixture thereof.

12. The liquid-crystalline medium according to claim 1, additionally comprising at least one compound of the formulae



in which

 $R^7$  and  $R^8$  are each, independently of one another, as defined for  $R^{11},\,R^{12}$  and  $R^{21},$  and

w and x are each, independently of one another, from 1 to 6.

13. The liquid-crystalline medium according to claim 2, comprising

10-40% by weight of at least one compound of formula I1,

5-30% by weight of at least one compound of formula I2, and

20-70% by weight of at least one compound of formula II.

- 14. An electro-optical display having active matrix addressing based on ECB effect or IPS effect, comprising as a dielectric, a liquid-crystalline medium according to claim 1.
- 15. An electro-optical display comprising, as a dielectric, a liquid-crystalline medium according to claim 1.
- 16. An electro-optical display comprising, as a dielectric, a liquid-crystalline medium according to claim 2.
- 17. An electro-optical display comprising, as a dielectric, a liquid-crystalline medium according to claim 3.

\* \* \* \* \*

# Generate Collection

L3: Entry 2 of 2 File: PGPB Feb 7, 2002 `

DOCUMENT-IDENTIFIER: US 20020014613 A1 TITLE: Liquid-crystalline medium

Detail Description Table CWU (16):

17 PCH-304FF 13.0% Clearing point [.degree. C.]: 70 PCH-502FF 8.0% .DELTA.n [589 nm, 20.degree. C.]: +0.0986 PCH-504FF 11.0% .DELTA..epsilon. [1 kHz, 20.degree. C.]: -3.2 CPY-3-02 10.0% .epsilon..sub..parallel. [1 kHz, 20.degree. C.]: 3.6 CPQIY-3-02 5.0% V.sub.o [V]: 2.12 CPQIY-3-04 5.0% .gamma..sub.1 [mPa .multidot. s, 20.degree. C.]: 116 CPY-2-02 9.0% BCH-32 8.0% CC-3-V1 8.0% CCH-35 5.0% CC-5-V 18.0%

Detail Description Table CWU (19):

20 PCH-304FF 11.0% Clearing point [.degree. C.]: 69.5 PCH-502FF 9.0% .DELTA.n [589 nm, 20.degree. C.]: +0.0952 PCH-504FF 16.0% .DELTA..epsilon. [1 kHz, 20.degree. C.] -3.6 CPQIY-3-02 8.0% .epsilon..sub..parallel. [1 kHz, 20.degree. C.]: 3.6 CPY-2-04 10.0% V.sub.o [V] 2.08 CPY-3-02 11.0% .gamma..sub.1 [mPa .multidot. s, 20.degree. C.] 120 CCPC-33 3.0% CC-3-V1 8.0% CCH-35 5.0% CC-5-V 19.0%

#### **End of Result Set**

**Generate Collection** Print

L6: Entry 2 of 2

File: PGPB

Feb 7, 2002

DOCUMENT-IDENTIFIER: US 20020014613 A1

TITLE: Liquid-crystalline medium

#### Detail Description Table CWU (18):

19 PCH-304FF 8.0% Clearing point [.degree. C.]: 70.0 PCH-502FF 10.0% .DELTA.n [589 nm, 20.degree. C.]: +0.1023 PCH-504FF 14.0% .DELTA..epsilon. [1 kHz, 20.degree. C.]: -3.3 CPY-3-02 12.0% .epsilon..sub..parallel. [1 kHz, 20.degree. C.]: 3.6 CQY-5-1 5.0% V.sub.o [V]: 2.14 CQY-3-04 5.0% .gamma..sub.1 [mPa .multidot. s, 20.degree. C.]: 104 CPY-3-04 12.0% BCH-32 9.0% CC-3-V1 10.0% CCH-35 5.0% CC-5-V 10.0%

```
Takeshita, Fusayuki; et al.
PA
     Chisso Corp., Japan; Ando, Tsugumichi; Matsui, Shuichi; Miyazawa,
     Kazutoshi; Takeuchi, Hiroyuki; Koizumi, Yasuyuki; Sekiguchi, Yasuko
SO
     PCT Int. Appl., 222 pp.
     CODEN: PIXXD2
DT
     Patent
     Japanese
LΑ
IC
     ICM C07C043-192
     ICS \( \)C07C043-225; C07C069-75; C07C069-753; C07C069-757; C07C069-76;
          C09K019-20; C09K019-30; C09K019-42; G02F001-13
CC
     75-11 (Crystallography and Liquid Crystals)
     Section cross-reference(s): 74
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GΙ
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$$R^1$$
  $X^1$   $X^2$   $X^2$   $X^3$   $X^3$   $X^4$   $Y^1$ 

The above liq. crystal compds. represented by general formula (I; R1, Y1 = C1-20 alkyl; X1, X2, X3 = a single bond, 1,2-ethylene, vinylene, CO2, CF2O or OCF2; A1, A2, A3, A4 = trans-1,4-cyclohexylene or optionally fluorinated or chlorinated 1,4-phenylene, at least one of A2, A3 and A4 being 2,3-difluoro-1,4-phenylene; m, n = 0 or 1) with the proviso that the elements constituting the compds. may be each replaced by isotopes thereof are prepd. Liq. crystal compns. contg. the compds. I and liq. crystal display devices made by using the compns. are claimed. These liq. crystal compds. are low in viscosity and high in dielec. anisotropy and have high specific resistance and high voltage retention, and are stable even when exposed to heat and UV rays. Thus, 2,3-difluoro-4-ethoxyphenol was

Ι

condensed with 2-fluoro-4-[2-(trans-4-propylcyclohexyl)ethyl]benzoic acid using DCC in the presence of 4-dimethylaminopyridine at room temp. overnight to give 2,3-difluoro-4-ethoxyphenyl 2-fluoro-4-[2-(trans-4-propylcyclohexyl)ethyl]benzoate (II), which showed phase transition temp. 65.4.degree. for C-N point and 141.3.degree. for N-I point. A liq. crystal compn. contg. 15 wt.\* II and a mother liq. crystal compn. (85 wt.\*) consisting of 4-butoxyphenyl 4-(trans-4-propylcyclohexyl)carboxybenzoate 27.6, 4-ethoxyphenyl 4-(trans-4-butylcyclohexyl)carboxybenzoate 20.7, 4-methoxyphenyl 4-(trans-4-pentylcyclohexyl)carboxybenzoate 20.7, 4-ethoxyphenyl 4-(trans-4-propylcyclohexyl)carboxybenzoate 17.2, and 4-ethoxyphenyl 4-(trans-4-pentylcyclohexyl)carboxybenzoate 13.8 wt.\* showed clearing point 129.4.degree. and dielec. anisotropy (.DELTA..epsilon.) -4.17 as compared to 74.6.degree. and 0.0, resp., for the mother liq. crystal compn.

- ST liq crystal compn display device; phenyl cyclohexylethylbenzoate prepn liq crystal
- IT Liquid crystal displays Liquid crystals

```
Liquid crystals
        (prepn. of liq. crystal compds., liq. crystal compns. contg. the
        compds., and liq. crystal display devices made by using them)
                               38690-77-6
                                             40817-08-1
                                                          41122-71-8
IT
     22692-80-4
                  38444-13-2
                  50649-60-0
                                                          56131-49-8
     50649-59-7
                               52709-83-8
                                             56131-48-7
                                                          58743-75-2
     57202-28-5
                  57202-30-9
                               57202-37-6
                                             57202-38-7
                  61203-99-4
                                                          63221-88-5
     59855-05-9
                               61204-01-1
                                             61204-03-3
                  64240-64-8
                               64835-59-2
                                             67589-39-3
                                                          67589-41-7
     63295-01-2
     67589-46-2
                  67589-47-3
                               67589-52-0
                                                          68400-50-0
                                             67589-53-1
                  72928-54-2
     70567-18-9
                               74305-48-9
                                             75941-46-7
                                                          75941-48-9
                  75941-51-4
                                             75941-90-1
     75941-50-3
                               75941-52-5
                                                          76802-59-0
                               79709-84-5
     76802-61-4
                  79319-27-0
                                             79832-86-3
                                                          79912-85-9
     79945-42-9
                  80944-44-1
                               80955-71-1
                                             81701-13-5
                                                          81711-13-9
     81793-57-9
                  81793-59-1
                               81936-32-5
                                             82832-27-7
                                                          82832-33-5
                               82985-80-6
     82832-34-6
                  82832-57-3
                                             82991-48-8
                                                          83242-83-5
     84540-37-4
                  84655-98-1
                               84656-75-7
                                             84656-77-9
                                                          84656-92-8
     85312-59-0
                  86377-38-0
                               86579-52-4
                                             86778-48-5
                                                          86786-89-2
     86840-79-1
                                             88038-92-0
                  87073-93-6
                               87260-24-0
                                                          88416-69-7
     88416-84-6
                  88416-89-1
                               88639-41-2
                                             88878-50-6
                                                          89129-90-8
     92263-41-7
                  93743-04-5
                               95495-03-7
                                             95495-15-1
                                                          95495-17-3
     95495-18-4
                  95906-29-9
                               95906-34-6
                                             96624-41-8
                                                          96624-52-1
     97398-80-6
                  98321-58-5
                               98495-10-4
                                             98495-11-5
                                                          98495-16-0
     98495-17-1
                  100497-33-4
                                100980-86-7
                                               101478-47-1
                                                             101559-74-4
     102714-92-1
                   102714-93-2
                                 102714-95-4
                                                106021-42-5
                                                              107215-66-7
     107215-73-6
                   107215-74-7
                                  107392-35-8
                                                112026-68-3
                                                              114291-10-0
     116090-24-5
                   116090-26-7
                                 116090-30-3
                                                116090-32-5
                                                              116090-33-6
     116090-34-7
                   116090-36-9
                                 116090-37-0
                                                116903-46-9
                                                              116903-47-0
     116903-49-2
                   117923-23-6
                                 117943-37-0
                                                118164-50-4
                                                              118164-51-5
     120893-64-3
                   121218-79-9
                                 121218-80-2
                                                121218-90-4
                                                              121219-85-0
     122412-08-2
                   123560-47-4
                                 123560-48-5
                                                123560-54-3
                                                              123560-56-5
                                 124728-81-0
     123787-66-6
                   123787-68-8
                                                124729-02-8
                                                              124770-58-7
     124770-60-1
                   124794-57-6
                                 129738-34-7
                                                129738-42-7
                                                              130746-72-4
     131819-23-3
                   131819-24-4
                                 131819-25-5
                                                132123-39-8
                                                              132123-43-4
     132123-45-6
                   132123-46-7
                                 133914-49-5
                                                133914-50-8
                                                              133937-72-1
                                                              136922-42-4
     134412-17-2
                   134412-18-3
                                 135734-59-7
                                                135734-60-0
     137019-95-5
                   137529-41-0
                                 137529-43-2
                                                137529-56-7
                                                              139136-72-4
     139420-31-8
                   140212-76-6
                                 145131-04-0
                                                145131-05-1
                                                              145305-20-0
                                                              148462-52-6
     145918-41-8
                   146781-29-5
                                 146781-31-9
                                                148462-51-5
     153429-48-2
                   155041-85-3
                                 155905-85-4
                                                157248-24-3
                                                              162744-15-2
     173306-39-3
                   173476-21-6
                                 173476-46-5
                                                174350-05-1
                                                              174350-07-3
     174350-08-4
                   175859-23-1
                                 175859-24-2
                                                175859-25-3
                                                              175859-28-6
     175859-31-1
                   176176-33-3
                                 178689-87-7
                                                181943-57-7
                                                              183145-19-9
     184161-94-2
                   186320-72-9
                                 196870-32-3
                                                197012-63-8
                                                              197012-64-9
     197012-65-0
                   197012-66-1 197012-67-2
                                            197012-68-3
     197012-69-4
                   197012-71-8
                                 197012-72-9
                                                197012-73-0
                                                              197012-75-2
     197012-76-3
                   197012-77-4
                                 197012-78-5
                                                197012-79-6
                                                              197012-83-2
```

```
197012-84-3
                   197012-85-4
                                 197012-86-5
                                               197012-87-6
     RL: DEV (Device component use); TEM (Technical or engineered material
     use); USES (Uses)
        (liq. crystal compn. contg.; prepn. of liq. crystal compds., liq.
        crystal compns. contg. the compds., and liq. crystal display devices
        made by using them)
                  197012-58-1 197012-59-2
IT
     197012-57-0
                                             197012-60-5
                  197012-62-7 197012-70-7 197012-74-1
     197012-61-6
     197012-80-9 197012-81-0 197012-82-1
     RL: DEV (Device component use); TEM (Technical or engineered material
     use); USES (Uses)
        (liq. crystal compn.; prepn. of liq. crystal compds., liq. crystal
        compns. contg. the compds., and liq. crystal display devices made by
        using them)
IT
     197012-44-5P 197012-45-6P
                                 197012-46-7P
                                                197012-47-8P
                   197012-49-0P
                                   197012-50-3P 197012-51-4P
     197012-48-9P
     197012-52-5P
                   197012-53-6P
                                   197012-54-7P
                                                 197012-55-8P
                                                                 197012-56-9P
     RL: DEV (Device component use); SPN (Synthetic preparation); TEM
     (Technical or engineered material use); PREP (Preparation); USES (Uses)
        (prepn. of liq. crystal compds., liq. crystal compns. contg. the
        compds., and liq. crystal display devices made by using them)
IT
     74-96-4, Ethyl bromide 75-15-0, Carbon disulfide, reactions
     Trimethyl borate 1031-15-8, Methyltriphenylphosphonium chloride
     4009-98-7, Methoxymethyltriphenylphosphonium chloride 4746-97-8,
     1,4-Cyclohexanedione monoethylene ketal 6418-38-8, 2,3-Difluorophenol
     6793-92-6, Benzyl 4-Bromophenyl ether 14938-35-3, 4-Pentylphenol
     65355-32-0, trans-4-(trans-4-Propylcyclohexyl)cyclohexanecarboxylic acid
     69891-92-5, 2-(1,3-Dioxan-2-yl)ethyltriphenylphosphonium bromide
     79832-89-6, 4-(trans-4-n-Pentylcyclohexyl)phenyl bromide
                                                                81936-33-6,
     4-(trans-4-Propylcyclohexyl)phenol
                                        86579-53-5, 4-(trans-4-
     Propylcyclohexyl) bromobenzene
                                     132248-38-5
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (prepn. of liq. crystal compds., liq. crystal compns. contg. the
        compds., and liq. crystal display devices made by using them)
     51171-73-4P, 4-(4-Hydroxyphenyl)cyclohexanone
TT
                                                     121219-07-6P,
     2,3-Difluoro-1-ethoxybenzene
                                   121537-69-7P 126163-56-2P,
                                 197012-88-7P, 4-(trans-4-n-
     2,3-Difluoro-4-ethoxyphenol
     Pentylcyclohexyl) phenylmagnesium bromide 197012-89-8P,
     4-(trans-4-n-Pentylcyclohexyl)phenyldithiocarboxylic acid
                                                                 197012-90-1P
     197012-91-2P, 4-(trans-4-Propylcyclohexyl)phenyldithiocarboxylic acid
     197012-92-3P, 4-(trans-4-Propylcyclohexyl)phenylthiocarbonyl chloride
     197012-93-4P, 4-Pentylphenyl 4-(trans-4-propylcyclohexyl)thiobenzoate
                   197012-95-6P, 4-Propylphenylthiocarbonyl chloride
     197012-94-5P
     197012-96-7P
                    197012-97-8P
                                   197012-98-9P
                                                  197012-99-0P
                                                                 197013-00-6P
     197013-01-7P
                    197013-02-8P
                                   197013-03-9P
                                                  197013-04-0P
                                                                 197013-05-1P
     197013-06-2P
                   197013-07-3P
                                   197013-08-4P
                                                  197013-09-5P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
     (Reactant or reagent)
        (prepn. of liq. crystal compds., liq. crystal compns. contg. the
        compds., and liq. crystal display devices made by using them)
IT
     197012-67-2
    RL: DEV (Device component use); TEM (Technical or engineered material
     use); USES (Uses)
        (liq. crystal compn. contg.; prepn. of liq. crystal compds., liq.
        crystal compns. contg. the compds., and liq. crystal display devices
       made by using them)
RN
     197012-67-2 CAPLUS
CN
    Benzene, 1-[difluoro[4-(4-methylcyclohexyl)phenyl]methoxy]-4-ethoxy-2,3-
    difluoro-, trans- (9CI) (CA INDEX NAME)
```

IT 197012-59-2 197012-70-7 197012-74-1 197012-80-9 197012-82-1

RL: DEV (Device component use); TEM (Technical or engineered material use); USES (Uses)

(liq. crystal compn.; prepn. of liq. crystal compds., liq. crystal compns. contg. the compds., and liq. crystal display devices made by using them)

RN 197012-59-2 CAPLUS

CN Benzoic acid, 4-[[(4-butylcyclohexyl)carbonyl]oxy]-, 4-ethoxyphenyl ester, trans-, mixt. with trans-4-butoxyphenyl 4-[[(4-propylcyclohexyl)carbonyl]oxy]benzoate, trans-1-[difluoro[4-(4-pentylcyclohexyl)phenyl]methoxy]-4-ethoxy-2,3-difluorobenzene, trans-4-ethoxyphenyl 4-[[(4-pentylcyclohexyl)carbonyl]oxy]benzoate, trans-4-ethoxyphenyl 4-[[(4-propylcyclohexyl)carbonyl]oxy]benzoate and trans-4-methoxyphenyl 4-[[(4-pentylcyclohexyl)carbonyl]oxy]benzoate (9CI) (CA INDEX NAME)

CM 1

CRN 197012-45-6 CMF C26 H32 F4 O2

Relative stereochemistry.

CM 2

CRN 70592-21-1 CMF C26 H32 O5

CRN 70567-30-5 CMF C27 H34 O5

Relative stereochemistry.

CM 4

CRN 70567-20-3 CMF C26 H32 O5

CRN 70567-14-5 CMF C27 H34 O5

Relative stereochemistry.

CM 6

CRN 70567-12-3 CMF C25 H30 O5

Relative stereochemistry.

RN 197012-70-7 CAPLUS

CN 1,1'-Biphenyl, 3,4-difluoro-4'-(4-pentylcyclohexyl)-, trans-, mixt. with [trans(trans)]-1,2-difluoro-4-(4'-pentyl[1,1'-bicyclohexyl]-4-yl)benzene, trans-1-[difluoro[4-(4-pentylcyclohexyl)phenyl]methoxy]-4-ethoxy-2,3-difluorobenzene, [trans(trans)]-1,2-difluoro-4-(4'-propyl[1,1'-bicyclohexyl]-4-yl)benzene, trans-3,4-difluoro-4'-(4-propylcyclohexyl)-1,1'-biphenyl, [trans(trans)]-1,2-difluoro-4-[4-[2-(4-propylcyclohexyl)ethyl]cyclohexyl]benzene, [trans(trans)]-4-(4'-ethyl[1,1'-bicyclohexyl]-4-yl)-1,2-difluorobenzene, trans-4'-(4-ethylcyclohexyl)-3,4-difluoro-1,1'-biphenyl and [trans(trans)]-4-[4-[2-(4-ethylcyclohexyl)ethyl]cyclohexyl]-1,2-difluorobenzene (9CI) (CA INDEX NAME)

CM 1

CRN 197012-45-6

Relative stereochemistry.

CM 2

CRN 134412-18-3 CMF C20 H22 F2

Relative stereochemistry.

CM 3

CRN 134412-17-2 CMF C23 H28 F2

CRN 118164-51-5 CMF C23 H34 F2

Relative stereochemistry.

Me (CH<sub>2</sub>) 
$$\frac{1}{4}$$

CM 5

CRN 118164-50-4 CMF C20 H28 F2

Relative stereochemistry.

CM 6

CRN 117943-37-0 CMF C23 H34 F2

CRN 117923-19-0 CMF C22 H32 F2

Relative stereochemistry.

CM 8

CRN 85312-59-0 CMF C21 H24 F2

Relative stereochemistry.

CM 9

CRN 82832-57-3 CMF C21 H30 F2

Relative stereochemistry.

RN 197012-74-1 CAPLUS

CN Cyclohexanecarboxylic acid, 4-butyl-, 4-ethoxyphenyl ester, trans-, mixt. with trans-4-butoxyphenyl 4-propylcyclohexanecarboxylate, trans-1-butoxy-4-(4-propylcyclohexyl)benzene, trans-1-[difluoro[4-(4-pentylcyclohexyl)phenyl]methoxy]-4-ethoxy-2,3-difluorobenzene, [1.alpha.,4.beta.[E(trans)]]-4-ethoxy-2,3-difluorophenyl 4-[2-(4-pentylcyclohexyl)ethenyl]cyclohexanecarboxylate, trans-4-ethoxyphenyl 4-pentylcyclohexanecarboxylate, trans-4-ethoxyphenyl 4-propylcyclohexanecarboxylate, trans-1-ethoxy-4-(4-propylcyclohexyl)benzene and trans-4-methoxyphenyl 4-pentylcyclohexanecarboxylate (9CI) (CA INDEX NAME)

CM 1

CRN 197012-52-5 CMF C28 H40 F2 O3

Relative stereochemistry.

Double bond geometry as shown.

CM 2

CRN 197012-45-6 CMF C26 H32 F4 O2 Relative stereochemistry.

CM 3

CRN 80944-44-1 CMF C17 H26 O

Relative stereochemistry.

CM 4

CRN 79709-84-5 CMF C19 H30 O

Relative stereochemistry.

CM 5

CRN 67589-53-1 CMF C20 H30 O3

CRN 67589-52-0 CMF C19 H28 O3

Relative stereochemistry.

CM 7

CRN 67589-47-3 CMF C19 H28 O3

Relative stereochemistry.

CM 8

CRN 67589-41-7 CMF C20 H30 O3

CRN 67589-39-3 CMF C18 H26 O3

Relative stereochemistry.

RN 197012-80-9 CAPLUS

CN Cyclohexanecarboxylic acid, 4-butyl-, 4-ethoxyphenyl ester, trans-, mixt. with trans-4-butoxyphenyl 4-propylcyclohexanecarboxylate, trans-1-[difluoro[4-(4-pentylcyclohexyl)phenyl]methoxy]-4-ethoxy-2,3-difluorobenzene, trans-4-ethoxyphenyl 4-pentylcyclohexanecarboxylate, trans-4-ethoxyphenyl 4-propylcyclohexanecarboxylate and trans-4-methoxyphenyl 4-pentylcyclohexanecarboxylate (9CI) (CA INDEX NAME)

CM 1

CRN 197012-45-6 CMF C26 H32 F4 O2

Relative stereochemistry.

CM 3

CRN 67589-52-0 CMF C19 H28 O3

Relative stereochemistry.

CM

CRN 67589-47-3 CMF C19 H28 O3

CRN 67589-41-7 CMF C20 H30 O3

Relative stereochemistry.

CM 6

CRN 67589-39-3 CMF C18 H26 O3

Relative stereochemistry.

RN 197012-82-1 CAPLUS
CN Benzoic acid, 2-fluoro-4-[2-(4-propylcyclohexyl)ethyl]-,
4-ethoxy-2,3-difluorophenyl ester, trans-, mixt. with 4-butoxy-2,3difluoro-4'-pentyl-1,1'-biphenyl, 4-butoxy-2,3-difluoro-4'-propyl-1,1'biphenyl, 2',3'-difluoro-4,4''-dipentyl-1,1':4',1''-terphenyl,
2',3'-difluoro-4-heptyl-4''-pentyl-1,1':4',1''-terphenyl,
trans-1-[difluoro[4-(4-pentylcyclohexyl)phenyl]methoxy]-4-ethoxy-2,3difluorobenzene, 2',3'-difluoro-4-pentyl-4''-propyl-1,1':4',1''-terphenyl,
4-ethoxy-2,3-difluoro-4'-propyl-1,1'-biphenyl and 4-ethyl-2',3'-difluoro4''-propyl-1,1':4',1''-terphenyl (9CI) (CA INDEX NAME)

CM 1

CRN 197012-45-6 CMF C26 H32 F4 O2

Me 
$$(CH_2)^{\frac{1}{4}}$$

CRN 197012-44-5 CMF C26 H31 F3 O3

Relative stereochemistry.

CM 3

CRN 157248-28-7 CMF C19 H22 F2 O

CM 4

CRN 157248-27-6 CMF C21 H26 F2 O

$$n-BuO$$
 $F$ 
(CH<sub>2</sub>)<sub>4</sub>-Me

CRN 157248-25-4 CMF C23 H22 F2

CM 6

CRN 157248-24-3 CMF C17 H18 F2 O

CM 7

CRN 121235-87-8 CMF C30 H36 F2

$$Me^{-(CH_2)}4$$
  $F$   $F$   $(CH_2)_6-Me$ 

CM 8

CRN 121218-79-9 CMF C26 H28 F2

$$n-Pr$$
 $F$ 
(CH<sub>2</sub>)<sub>4</sub>-Me

CRN 121218-76-6 CMF C28 H32 F2

$$Me^{-(CH_2)}\frac{1}{4}-Me^{-(CH_2)}\frac{1}{4}-Me^{-(CH_2)}\frac{1}{4}$$

IT 197012-45-6P 197012-51-4P

RL: DEV (Device component use); SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses) (prepn. of liq. crystal compds., liq. crystal compns. contg. the compds., and liq. crystal display devices made by using them)

RN 197012-45-6 CAPLUS

CN Benzene, 1-[difluoro[4-(trans-4-pentylcyclohexyl)phenyl]methoxy]-4-ethoxy2,3-difluoro- (9CI) (CA INDEX NAME)

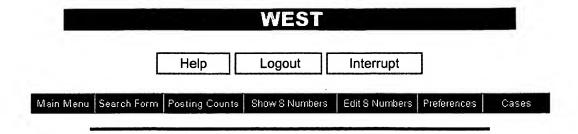
Relative stereochemistry.

Me 
$$(CH_2)^{\frac{1}{4}}$$

RN 197012-51-4 CAPLUS

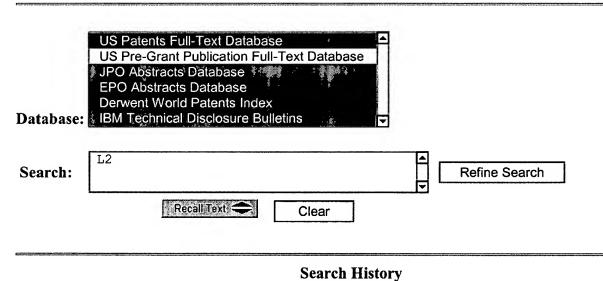
CN Benzene, 1-[difluoro[4-(4'-propyl[1,1'-bicyclohexyl]-4-yl)phenyl]methoxy]-4-ethoxy-2,3-difluoro-, [trans(trans)]- (9CI) (CA INDEX NAME)

.



#### Search Results -

Term	Documents
US-6319570-\$	0
US-6319570-B1.DWPI,EPAB,JPAB,USPT.	2
US-6190576-\$	0
US-6190576-B1.DWPI,EPAB,JPAB,USPT.	2
((US-6319570-\$.DID.) OR (US-6190576-\$.DID.)).USPT,JPAB,EPAB,DWPI,TDBD.	3
(US-6319570-\$.DID. OR US-6190576-\$.DID.).USPT,JPAB,EPAB,DWPI,TDBD.	3



DATE: Friday, March 21, 2003 Printable Copy Create Case

	Set Name Query side by side				
DB=U	SPT,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ				
<u>L2</u>	US-6319570-\$.did. or us-6190576-\$.did.	3	<u>L2</u>		
<u>L1</u>	US-6395353-\$.did. or JP-11029581-\$.did. or WO-9736847-\$.did.	6	<u>L1</u>		

#### **END OF SEARCH HISTORY**



JS006395353B2

# (12) United States Patent Yanai et al.

(10) Patent No.:

US 6,395,353 B2

(45) Date of Patent:

May 28, 2002

## (54) LIQUID CRYSTAL COMPOSITION AND LIQUID CRYSTAL DISPLAY ELEMENT

- (75) Inventors: Motoki Yanai; Yasuhiro Kubo; Etsuo Nakagawa, all of Ichihara (JP)
- (73) Assignees: Chisso Corporation, Osaka; Chisso Petrochemical Corporation, Tokyo, both of (JP)
- (\*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.
- (21) Appl. No.: 09/808,019(22) Filed: Mar. 15, 2001
- (30) Foreign Application Priority Data

428/1.

(56) References Cited

## U.S. PATENT DOCUMENTS

5,993,690 A 11/1999 Kondo et al. .......... 252/299.6

#### FOREIGN PATENT DOCUMENTS

EP 1114825 \* 7/2001 JP 2001-31685 \* 2/2001 JP 2001-31972 \* 2/2001

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#### (57) ABSTRACT

The present invention provides a liquid crystal composition comprising the compounds represented by the following formulas (I) and (II)

$$R^{1} - \left(Z^{1} - A^{1}\right)_{\overline{m}} - Z^{2} - \left(II\right)$$

$$R^{1} - \left(II\right)$$

$$R^{2} - \left(III\right)$$

$$R^{1}$$
  $(A^{2}$   $Z^{3})_{a}$   $A^{3}$   $Z^{4}$   $R^{2}$ 

wherein each  $R^1$  independently represents an alkyl group having 1–10 carbon atoms or an alkenyl group having 2–10 carbon atoms; each  $R^2$  independently represents an alkyl or alkoxy group having 1–10 carbon atoms or an alkenyl group having 2–10 carbon atoms;  $Z^1$  and  $Z^2$  represent —CH<sub>2</sub>SiH<sub>2</sub>—, a single bond or —CH<sub>2</sub>CH<sub>2</sub>—, with the proviso that one of  $Z^1$  and  $Z^2$  represents —CH<sub>2</sub>SiH<sub>2</sub>— and the other represents a single bond or —CH<sub>2</sub>CH<sub>2</sub>—;  $A^1$ ,  $A^2$  and  $A^3$  each independently represent a ring including aromatic, alicyclic and heterocyclic groups; m and n are each independently 0 or 1; and  $Z^3$  and  $Z^4$  each independently represent a single bond, —CH<sub>2</sub>CH<sub>2</sub>—, —CF<sub>2</sub>O— or —OCF<sub>2</sub>—. The composition has a suitable  $\Delta n$ , a low viscosity, a largely negative  $\Delta e$  and a broad nematic liquid crystal phase range while satisfying various properties required for liquid crystal compositions for AM-LCD.

#### 5 Claims, No Drawings

<sup>\*</sup> cited by examiner

#### FIELD OF THE INVENTION

The present invention relates to a nematic liquid crystal composition having a negative dielectric anisotropy and to a liquid crystal display element containing the composition. More particularly, it relates to a liquid crystal composition having a negative dielectric anisotropy which is used for an active matrix liquid crystal display element, and to a liquid crystal display element containing the composition.

#### **BACKGROUND OF THE INVENTION**

A liquid crystal display (LCD) element consumes less <sup>15</sup> electric power, and can be downsized and save weight as compared with a cathode ray tube (CRT) display. Therefore, several kinds of liquid crystal display systems such as twist nematic (TN), super twist nematic (STN) and thin film transistor (TFT) modes have been put to practical use. <sup>20</sup> Among them, an active matrix liquid crystal display element (AM-LCD) has been the focus of interest as the most expected system because of its advanced colorization and minuteness.

A liquid crystal composition used in AM-LCD is required  $^{25}$  to have the following properties:

- 1) high voltage holding ratio to maintain high contrast of the liquid crystal display element,
- broad nematic phase range to cope with various 30 environments.
- 3) suitable refractive anisotropy ( $\Delta n$ ) in accordance with cell thickness, and
- 4) suitable threshold voltage in accordance with drive circuit.

A drive system of AM-LCD has mainly been TN mode in which twist angle of alignment of liquid crystal molecules between upper and lower electrode substrates is 90 degrees. However, it has a difficulty in applying for wide view displays because of the narrow view angle. Some modes for improving view angle have been proposed as follows:

- a) IPS display mode in which a liquid crystal display element shows homogeneous alignment of liquid crystal molecules when voltage is unimpressed and the molecules rotate at an angle of 45 to 90 degrees within the same plane when voltage is impressed (R. Kiefer, B. Weber, F. Windscheid and G. Baur, In-Plane Switching of Nematic Liquid Crystals, JAPAN DISPLAY '92, p. 547; and
- b) VA display mode in which a liquid crystal display solution element shows homeotropic alignment when voltage is unimpressed and then shows horizontal alignment in one direction when voltage is impressed (K. Ohmuro, S. Kataoka, T. Sasaki and Y. Koike, Development of Super-High-Image-Quality Vertical-Alignment-Mode LCD, SID 55 Digest, p. 845).

These display systems have characteristics of quick response and high contrast in addition to the wide view angle. Further, they have a remarkable characteristic that they can comprise a liquid crystal composition having a  $_{60}$  negative dielectric anisotropy ( $\Delta\epsilon$ ).

These drive systems utilize an electrically controlled birefringence mode. A product ( $\Delta$ n.d) of refractive anisotropy ( $\Delta$ n) and cell thickness (d) should be, for example,  $\Delta$ n.d=approx. 0.275  $\mu$ m so as to provide a suitable contrast. 65 Accordingly, a  $\Delta$ n value should be 0.05 to 0.13 so as to obtain a cell thickness of 2 to 6  $\mu$ m. Since response time is

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proportioned to viscosity of a liquid crystal composition (hereinafter referred to as  $\eta$ ), a liquid crystal composition is required to have a small  $\eta$ . Further, threshold voltage decreases as an absolute value of dielectric anisotropy (hereinafter referred to as  $\Delta \epsilon$ ) increases, and therefore, a liquid crystal composition is required to have a largely negative  $\Delta \epsilon$ .

Such a liquid crystal composition has been studied for various purposes, but is always required to be much more improved.

#### SUMMARY OF THE INVENTION

An object of the present invention is to provide a liquid crystal composition applicable for the display systems of the above a) and b), which can realize a wide view angle, a suitable  $\Delta n$ , and particularly a largely negative dielectric anisotropy, a broad range of nematic liquid crystal phase, a high voltage holding ratio and a low viscosity while satisfying various properties required for a liquid crystal composition for the AM-LCD.

The present inventors have studied liquid crystal compositions comprising various kinds of liquid crystalline compounds to solve the above problems, and found that the object can be achieved by a liquid crystal composition comprising several compounds as Components I to III, thus attaining the present invention.

The first liquid crystal composition of the present invention is described below in the items (1) and (2).

(1) A liquid crystal composition comprising Component I selected from the group consisting of the compounds represented by formula (I), Component II selected from the group consisting of the compounds represented by formula (II) and Component III selected from the group consisting of the compounds represented by formulas (III-1) and (III-2)

$$R^{1}$$
 $(Z^{1}$ 
 $A^{1}$ 
 $E$ 
 $E$ 
 $E$ 
 $(I)$ 

$$R^{1}$$
— $(A^{2}$ — $Z^{3})_{\overline{n}}$ — $A^{3}$ — $Z^{4}$ — $R^{2}$ 

(III-1) 
$$R^{1} - (COO)_{\overline{p}} - A^{4} - (COO)_{\overline{q}} - R^{3}$$
 (III-2)

$$R^{1}$$
— $A^{5}$ — $R^{3}$ 

wherein each R<sup>1</sup> independently represents an alkyl group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms; each R<sup>2</sup> independently represents an alkyl or alkoxy group having 1-10 carbon atoms, or an alkenyl group having 2-10 carbon atoms; Z<sup>1</sup> and Z<sup>2</sup> represent —CH<sub>2</sub>SiH<sub>2</sub>—, a single bond or —CH<sub>2</sub>CH<sub>2</sub>—, with the proviso that one of Z<sup>1</sup> and Z<sup>2</sup> represents —CH<sub>2</sub>SiH<sub>2</sub>— and

the other represents a single bond or -CH2CH2-; ring A1 represents a trans-1,4-cyclohexylene group, or a 1,4phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; m is 0 or 1, with the proviso that when m is 0, Z<sup>2</sup> represents -CH<sub>2</sub>SiH<sub>2</sub>-; Z<sup>3</sup> and Z<sup>4</sup> each independently represent a single bond, —CH<sub>2</sub>CH<sub>2</sub>—, —CF<sub>2</sub>O— or —OCF<sub>2</sub>—; rings A<sup>2</sup> and A<sup>3</sup> represent a tetrahydropyran-2,5-diyl group, a trans-1,4cyclohexylene group, a cyclohexa-1-ene-1,4-diyl group, or a 1,4-phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; n is 0 or 1; with the proviso that when n=0 and Z4 represents a single bond or -CH<sub>2</sub>CH<sub>2</sub>—, then ring A<sup>3</sup> represents a tetrahydropyran-2. 5-diyl group; when n=0 and Z<sup>4</sup> represents -CF<sub>2</sub>O or -OCF<sub>2</sub>—, then ring A<sup>3</sup> represents a trans-1,4cyclohexylene group or a cyclohexa-1-ene-1,4-diyl group; 15 when n=1 and Z<sup>3</sup> and Z<sup>4</sup> each independently represent a single bond or  $-CH_2CH_2$ —, then one of rings  $A^2$  and  $A^3$  represents a tetrahydropyran-2,5-diyl group; when n=1 and  $Z^3$  and/or  $Z^4$  represent  $-CF_2O$ — or  $-OCF_2$ —, then rings  $A^2$  and  $A^3$  represent a trans-1,4-cyclohexylene group, a 20 cyclohexa-1-ene-1,4-diyl group, or a 1,4-phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; each R3 independently represents an alkyl group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms, in which one -CH<sub>2</sub> may be substituted with -O-; ring A<sup>4</sup> represents a 1,4-phenylene group or a trans-1,4-cyclohexylene group; ring A<sup>5</sup> represents a trans-1,4-cyclohexylene group, or a 1,4-phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; p, q and s are each independently 0 or 1 with the proviso that (p+q) is 0 or 1.

(2) A liquid crystal composition as described in the above item (1), which comprises 3-80% by weight of Component I, 3-80% by weight of Component II and 1-80% by weight of Component III.

The second liquid crystal composition of the present invention is described below in the item (3).

(3) A liquid crystal composition as described in the above item (1) or (2), which further comprises Component IV selected from the group consisting of the compounds represented by formula (IV)

$$\mathbb{R}^4 - \left( Z^5 - A^6 \right)_1 - Z^6 - \mathbb{R}^5$$

wherein R<sup>4</sup> represents an alkyl group having 1-10 carbon 50 atoms or an alkenyl group having 2-10 carbon atoms, R<sup>5</sup> represents an alkyl or alkoxy group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms, Z<sup>5</sup> and Z<sup>6</sup> each independently represent a single bond or —CH<sub>2</sub>CH<sub>2</sub>—, ring A<sup>6</sup> represents a 1,4-phenylene group or 55 a trans-1,4-cyclohexylene group, and t is 0 or 1.

The third liquid crystal composition of the present invention is described below in the item (4).

(4) A liquid crystal composition as described in any one of the items (1) to (3), wherein the clearing point (Tc) is 60 60 to  $100^{\circ}$  C., the refractive anisotropy ( $\Delta$ n) is 0.06 to 0.12 and the dielectric anisotropy ( $\Delta$  $\epsilon$ ) is -6 to -1.

The liquid crystal display element of the present invention is described below in the item (5).

(5) A liquid crystal display element containing the liquid 65 R crystal composition as described in any one of the items (1) to (4).

## DETAILED DESCRIPTION OF THE INVENTION

Component I of the liquid crystal composition according to the present invention comprises the compounds represented by formula (I), wherein the clearing point (Tc) is in the range of -50 to  $100^{\circ}$  C., the refractive anisotropy ( $\Delta n$ ) is in the range of 0.05 to 0.170, and the dielectric anisotropy ( $\Delta \epsilon$ ) is in the range of -6 to -3. It is superior in heat stability and chemical stability, and plays a role of decreasing the threshold voltage of a liquid crystal composition for TFT which requires high reliability. However, when a composition having a negative  $\Delta \epsilon$  is prepared only from Component I, the composition may not have a high clearing point (Tc) or a suitable  $\Delta n$ , and compatibility of the composition at low temperature may be lowered.

Among the compounds represented by formula (I) as Component I, the following compounds are preferable. In the formulas,  $R^1$  and  $R^2$  each have the same meaning as defined above.

$$R^1$$
— $CH_2$ — $SiH_2$ — $R^2$  (I-2)

$$R^1$$
—CH<sub>2</sub>—SiH<sub>2</sub>— $R^2$ 
(I-3)

$$R^1$$
—CH<sub>2</sub>CH<sub>2</sub>—CH<sub>2</sub>—SiH<sub>2</sub>— $F$ 
(1-4)

$$R^{1}$$
—CH<sub>2</sub>—SiH<sub>2</sub>— $F$ 
(I-5)

$$R^1$$
—CH<sub>2</sub>CH<sub>2</sub>— $R^2$ 
(1-6)

$$R^1$$
—CH<sub>2</sub>—SiH<sub>2</sub>— $R^2$ 

$$R^1 \hspace{-1mm} \longleftarrow \hspace{-1mm} CH_2 \hspace{-1mm} - \hspace{-1mm} SiH_2 \hspace{-1mm} \longleftarrow \hspace{-1mm} CH_2 \hspace{-1mm} CH_2 \hspace{-1mm} - \hspace{-1mm} CH_2 \hspace{-1mm} - \hspace{-1mm} R^2$$

-continued

-continued

$$R^{1} \longrightarrow CH_{2} \longrightarrow SiH_{2} \longrightarrow R^{2}$$

$$(I-9)$$

$$R^{1} \longrightarrow CH_{2} \longrightarrow SiH_{2} \longrightarrow CH_{2}CH_{2} \longrightarrow R^{2}$$

$$(I-10)$$

$$R^{1} \longrightarrow CH_{2} \longrightarrow SiH_{2} \longrightarrow R^{2}$$

Component II comprises the compounds represented by formula (II), wherein the clearing point (Tc) is in the range of -20 to  $160^{\circ}$  C., the refractive anisotropy ( $\Delta n$ ) is in the range of 0.05 to 0.170, and the dielectric anisotropy ( $\Delta \epsilon$ ) is in the range of -7 to -3. It is superior in heat stability and chemical stability, and plays a role of decreasing the threshold voltage of a liquid crystal composition for TFT which requires high reliability. However, when a composition having a negative  $\Delta \epsilon$  is prepared only from Component II, compatibility of the composition at low temperature may be unfavorably lowered.

Among the compounds represented by formula (II) as Component II, the following compounds are preferable. In the formulas,  $R^1$  and  $R^2$  each have the same meaning as defined above.

-continued

-continued

(11-24)

(II-25)

(11-26)

(II-27)

(II-28)

(II-29)

(II-30)

(II-31)

(II-32)

(11-23)

65

65

-continued

(II-42)

-continued

(II-33)

R

CH<sub>2</sub>CH<sub>2</sub>

CH<sub>2</sub>CH<sub>2</sub>

$$(II-36)$$
 $(II-36)$ 
 $(II-37)$ 
 $(II-37)$ 
 $(II-38)$ 
 $(II-38)$ 
 $(II-38)$ 

(II-38)

$$R^{1} \longrightarrow CH_{2}CH_{2} \longrightarrow CH$$

(II-36)
$$CH_{2}CH_{2} \longrightarrow CH_{2}CH_{2} \longrightarrow R^{2}$$
(II-37)
$$R \longrightarrow CCF_{2} \longrightarrow R^{2}$$
(II-38)
$$CH_{2}CH_{2} \longrightarrow CCF_{2} \longrightarrow R^{2}$$
(II-40)
$$CH_{2}CH_{2} \longrightarrow CCF_{2} \longrightarrow R^{2}$$
(II-41)
$$R \longrightarrow CCF_{2} \longrightarrow R^{2}$$

Component III comprises the compounds represented by formulas (III-1) and (III-2).

The compound represented by formula (III-1) has a clearing point (Tc) of 20 to 80° C., a refractive anisotropy ( $\Delta n$ ) of 0.01 to 0.08 and a dielectric anisotropy ( $\Delta \epsilon$ ) of approx. -1 to 0 as well as a low viscosity. It is superior in heat stability, chemical stability and compatibility, and plays a role of reducing  $\Delta n$  and viscosity, and adjusting  $\Delta \epsilon$  of the composition.

The compound represented by formula (III-2) has a clearing point (Tc) of 140 to 260° C., a refractive anisotropy ( $\Delta n$ ) of 0.10 to 0.20 and a dielectric anisotropy ( $\Delta \epsilon$ ) of approx. 0. It is superior in heat stability, chemical stability and compatibility, and plays a role of adjusting  $\Delta \epsilon$  as well as elevating the clearing point (Tc) of the composition. That is, Component III can provide a composition having a welladjusted clearing point (Tc), refractive anisotropy (An) and dielectric anisotropy ( $\Delta \epsilon$ ), as well as a low viscosity and an excellent compatibility at low temperature.

Among the compounds represented by formulas (III-1) and (III-2) as Component III, the following compounds are preferable. In the formulas, R1 and R2 each have the same meaning as defined above.

Any combination of several kinds of compounds selected from Components I, II and III, respectively, can provide a liquid crystal composition for AM-LCD having a suitable An, a low viscosity, a largely negative dielectric anisotropy, a broad nematic liquid crystal phase range and a high voltage holding ratio (VHR). Further, it can provide a composition having a clearing point (Tc) of 60 to 100° C., a refractive anisotropy ( $\Delta n$ ) of 0.06 to 0.12, a dielectric anisotropy ( $\Delta \epsilon$ ) of -6 to -1, a low viscosity and a broad nematic liquid crystal phase range.

Component IV comprises the compounds represented by formula (IV), which has a clearing point (Tc) of -20 to 180° C., a refractive anisotropy (An) of 0.06 to 0.21 and a dielectric anisotropy ( $\Delta \epsilon$ ) of approx. -7 to -3. It is superior in heat stability and chemical stability, and plays a role of decreasing threshold voltage of a liquid crystal composition 40 for TFT which requires high reliability.

Any combination of several kinds of compounds selected from Components I to IV, respectively, can provide the present liquid crystal composition for AM-LCD having a suitable An, a largely negative dielectric anisotropy, a low viscosity, a broad nematic liquid crystal phase range and a high voltage holding ratio (VHR).

In the liquid crystal composition of the present invention, the amount of Component I is preferably from 3 to 80% by amount is less than 3% by weight, the liquid crystal composition may unfavorably have the small absolute value of  $\Delta \epsilon$  (negative value) and the elevated threshold voltage. If the amount is more than 80% by weight, the compatibility at low temperature may unfavorably be lowered.

The amount of Component II is preferably from 3 to 80% by weight. More preferably it is 5 to 75% by weight. If the amount is less than 3% by weight, the liquid crystal composition may unfavorably have the small absolute value of weight, the compatibility at low temperature may unfavorably be lowered.

The amount of Component III is preferably 80% by weight or less. If the amount is more than 80% by weight, the liquid crystal composition may unfavorably have the 65 small absolute value of  $\Delta \epsilon$  (negative value) and the elevated threshold voltage.

The compounds contained as components in the liquid crystal composition of the present invention can be synthesized by the methods described in the following references.

Regarding the compounds represented by formula (III-1) as Component III, JP-A 59-70624 and JP-A 60-16940 describe methods for preparation of the compounds represented by formula (III-1-1), and JP-A 54-27546 describes a method for preparation of the compounds represented by formula (III-1-4), respectively.

Regarding the compounds represented by formula (III-2) as Component III, JP-A 57-165328 describes a method for preparation of the compounds represented by formula in (III-2-1).

Regarding the compounds represented by formula (II) as Component II, the compounds having a pyran ring represented by formulas (II-1) to (II-20) can be prepared by hydrosilylation (G. A. Kraus et al., J. Org. Chem., 46, 2417 (1981), G. A. Kraus et al., J. Chem. Soc., Chem. Commun., 1568, (1986)) of the compounds obtained from aldehyde derivatives and bromoacetic esters by Refomatsky reaction (M. W. RATHKE et al., J. O. C., 35 (11), 3966 (1970), J. F. RUPPERT et al., J. O. C., 39(2), 269 (1974)), the method of P. PICARD et al. (Synthesis, 550 (1981)), the method of Yamaguchi et al. (Tetrahedron Lett., 25(11), 1159 (1984)) and the like.

Regarding the compounds represented by formulas (II-21) to (II-43), the compounds represented by formulas (II-22), (II-23) and (II-33) to (II-43) which have  $-OCF_2$ as a connecting group can be obtained by reacting carboxylic acid derivatives with phenols or alcohols, carrying out condensation reaction by dehydration to synthesize compounds having an ester group as a connecting group, and then reacting the resultant ester derivatives with a publicly known sulfurization agent such as Lawson's reagent to synthesize compounds having a thiocarbonyl group, followed by fluorinating the compounds using fluorinating agents such as hydrogen fluoride pyridine (M. Kuroboshi et al., Chem. Lett., 827, 1992) and diethylaminosulfur trifluoride (William H. Bunnelle et al., J. Org. Chem. 1990, 55, 768)

The compounds represented by formulas (II-21), (II-24) to (II-32) can be similarly synthesized.

JP-A 6-228037 describes a method for preparation of the compounds represented by formula (IV) as Component IV.

The compounds represented by formula (I) as Component I can be synthesized by the same procedure as the compounds represented by formula (IV), and a preparation method of compounds having a silicon bond between rings weight. More preferably it is 5 to 75% by weight. If the 50 is described in WO 97/05144. Thus, each of the compounds as the component of the present liquid crystal composition can be synthesized according to the prior art.

The liquid crystal compositions of the present invention can be prepared by conventional methods per se. Typically, various components are mixed and dissolved with each other at high temperature.

The liquid crystal compositions of the present invention can also be used for guest-host (GH) mode displays with the addition of dichromatic dyes such as merocyanine, styryl, Δε (negative value). If the amount is more than 80% by 60 azo, azomethine, azoxy, quinophthalone, anthraquinone and tetrazine. The liquid crystal compositions of the present invention can also be used for NCAP which is produced by microencapsulating nematic liquid crystals, or polymer dispersed liquid crystal display (PDLCD) element which is typified by the polymer network liquid crystal display (PNLCD) element in which a three-dimensional matrix is formed in liquid crystals. The compositions can also be used with the addition of at least one chiral compound. In addition, the compositions can also be used for electrically controlled birefringence (ECB) mode or dynamic scattering (DS) mode displays.

The present invention will be described in detail by the following examples, but not limited thereto. In examples and comparative examples, all the composition ratios are indicated in terms of percentage by weight and the compounds used are represented by symbols as defined in Table 1. Property data of the liquid crystal compositions are shown in terms of clearing point (Tc), lower temperature limit of the nematic liquid crystal phase  $(T_L)$ , refractive anisotropy at 25° C.  $(\Delta e)$ , viscosity at

20° C.  $(\eta_{20})$  and voltage holding ratio at 25° C. and 80° C. (VHR(25° C.) and VHR(80° C.)).  $T_L$  was determined by the liquid crystal phase after the composition was allowed to stand for 30 days in the respective freezers at 0° C., -10° C., -20° C., -30° C. and -40° C.  $\Delta \epsilon (=\epsilon_{PA}-\epsilon_{PE})$  is obtained by measuring  $\epsilon_{PA}$  (dielectric constant in the direction parallel to symmetrical axis) and  $\epsilon_{PE}$  (dielectric constant in the direction perpendicular to symmetrical axis) using homeotropically aligned cells and homogeneously aligned cells. Voltage holding ratios (VHR) at 25° C. and 80° C. were measured by an area method using TN cell, in which a holding period was 16.6 msec and PIA-5210 manufactured by Chisso Corporation was used as an aligning film.

	TABL	E 1	
Method for	Designating Com	pounds by Using Symbols	
<del></del> F	<del>(</del>	$$ $Z_{\overline{n}}$ $(A_{\overline{n}})$ $X$	
1) Left Terminal Group R—	Symbol 3) Bor	nding Group —Z <sub>1</sub> —, —Z <sub>n</sub>	— Symbol
C <sub>n</sub> H <sub>2n+1</sub> — C <sub>n</sub> H <sub>2n+1</sub> OC <sub>m</sub> H <sub>2m</sub> — CH <sub>2</sub> =CH— CH <sub>2</sub> =CHC <sub>n</sub> H <sub>2n</sub> — CH <sub>2</sub> =CHC <sub>n</sub> H <sub>2n</sub> — C <sub>n</sub> H <sub>2n+1</sub> CH=CHC <sub>m</sub> H <sub>2m</sub> —	n- —CH <sub>2</sub> nOm- —SiH V- —C <sub>2</sub> H Vn- —COX nVm- —CF <sub>2</sub>	 	1 Si 2 E CF2O OCF2
2) Ring Structure —(A <sub>1</sub> )—, -	—(A <sub>n</sub> )— Symbol	4) Right Terminal Grou	p-X Symbol
<del></del>	Н	C <sub>n</sub> H <sub>2n+1</sub>	-0
	В	—OC <sub>n</sub> H <sub>2n+1</sub>	—On
F	B(F)	—СООСН <sub>3</sub>	—ЕМе
F	B(2F,3F)	—C <sub>n</sub> H <sub>2n</sub> OC <sub>m</sub> H <sub>2m+1</sub>	-лОm
<b>—</b>	Dh		
	Ch	i .	
5) Examples of Designation			

Example 1: V2-H1SiB(2F,3F)-O2

TABLE 1-continued

	Method for	Designating	Compounds b	v Using S	vmbols
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$$CH_2 = CHC_2H_4 - CH_2 - SiH_2 - F$$

$$CH_2 - SiH_2 - CH_2CH_3$$

Example 2: 101-HBBH-4

JP-A 6–228037 discloses an example of compounds having a negative  $\Delta \epsilon$  and compositions comprising them. Compositions A and C, which were disclosed in Examples 26 and 27 of the gazette, respectively, were prepared to measure the above physical properties.

#### Comparative Example 1 (Composition A)

3-HBB(2F,3F)-1O1	10.0%	
ZLI-1132	90.0%	

(Commercially available liquid crystal compound produced by Merck)

Tc=74.9° C.

 $T_z < -20^{\circ} \text{ C.}$ 

Δn=0.139

Δε=9.7

η<sub>20</sub>=29.2 mpa·s

VHR(25° C.)=95.8%

VHR(80° C.)=48.5%

The composition of Comparative Example 1 has a positive  $\Delta \epsilon$  and a remarkably low voltage holding ratio (VHR).

## Comparative Example 2 (Composition C)

5-HHB(2F,3F)-1O1	10.0%
ZLI-1132	90.0%

Tc=77.0° C.

T<sub>L</sub><-20° C.

Δn=0.133

Δε=9.7

 $\eta_{20}$ =29.1 mPa·s

VHR(25° C.)=96.1%

VHR(80° C.)=48.7%

The composition of Comparative Example 2 has a positive  $\Delta \epsilon$  and a remarkably low voltage holding ratio (VHR).

Since the compositions of Comparative Examples 1 and 2 contain a large amount of compounds having a cyano group, the voltage holding ratio (VHR) of the composition becomes 65 so small that the composition cannot apply for AM-LCD, and  $\Delta \epsilon$  is a positive value.

#### **EXAMPLE 1**

The composition comprising the following components was prepared.

	Component I	
	3-H1SiB(2F,3F)-O2	6.0%
	5-H1SiB(2F,3F)-O2	5.0%
	3-HH1SiB(2F,3F)-1	5.0%
30	2-HH1SiB(2F,3F)-O2	12.0%
	3-HH1SiB(2F,3F)-O2	9.0%
	3-HH1SiB(2F,3F)-O3	5.0%
	3-HB1SiB(2F,3F)-O2	3.0%
	Component II	
35	3-DhB(2F,3F)-O2	5.0%
	3-HDhB(2F,3F)-O2	10.0%
	5-HDhB(2F,3F)-O2	10.0%
	5-HHCF2OB(2F,3F)-O2	10.0%
	3-HHOCF2B(2F,3F)-O1	5.0%
	3-HB(2F,3F)OCF2B(2F,3F)-O1	5.0%
40	Component III	
	3-НН-4	4.0%
	3-HB-O2	3.0%
	3-НН-ЕМе	3.0%

The composition had the following physical properties:

Tc=75.0° C.

T<sub>r</sub><-20° C.

Δn=0.096

50

 $\Delta \epsilon = -4.6$ 

η<sub>20</sub>=37.8 mPa·s

VHR(25° C.)=99.1%

VHR(80° C.)=98.1%

The composition of Example 1 has an especially largely negative Δε and a very large voltage holding ratio (VHR) as 60 compared with those of Comparative Examples 1 and 2. Accordingly, it is suitable for the display systems of the above a) and b).

#### **EXAMPLE 2**

Component I			Component I	
3-H1SiB(2F,3F)-O2	12.0%	5	3-H1SiB(2F,3F)-O2	9.0%
5-H1SiB(2F,3F)-O2	12.0%		5-H1SiB(2F,3F)-O2	9.0%
2-HH1SiB(2F,3F)-O2	15.0%		3-HH1SiB(2F,3F)-1	6.0%
3-HH1SiB(2F,3F)-O2	13.0%		2-HH1SiB(2F,3F)-O2	15.0%
3-HH1SiB(2F,3F)-O3	13.0%		3-HH1SiB(2F,3F)-O2	13.0%
3-H1SiHB(2F,3F)-O2	3.0%		3-HH1SiB(2F,3F)-O3	13.0%
3-H1SiBB(2F,3F)-O2	3.0%	10	3-HB1SiB(2F,3F)-O2	3.0%
Component II			3-H1SiBB(2F,3F)-O2	3.0%
			Component II	
3-HDhB(2F,3F)-O2	4.0%		<del></del>	
5-HDhB(2F,3F)-O2	4.0%		3-BDhB(2F,3F)-O2	6.0%
3-BDhB(2F,3F)-O2	3.0%		5-HHCF2OB(2F,3F)-O2	3.0%
Component III		15	5-HBCF2OB(2F,3F)-O2	3.0%
			3-HB(2F,3F)OCF2B(2F,3F)-O1	4.0%
3-HB-O2	4.0%		Component III	
3-HHB-1	8.0%			
3-HHB-O1	6.0%		3-HB-O2	5.0%
			3-HHB-3	8.0%

The composition had the following physical properties:

Tc=60.1° C.

T<sub>L</sub><-20° C.

Δn=0.098

Δε=-3.6

η<sub>20</sub>=31.0 mPa·s

VHR(25° C.)=99.2%

VHR(80° C.)=98.4%

#### **EXAMPLE 3**

The composition comprising the following components was prepared.

The composition had the following physical properties:

Tc=60.2° C.

 $T_L < -20^{\circ} \text{ C}.$ 

Δn=0.100

 $\Delta \epsilon = -3.9$ 

η<sub>20</sub>=30.0 mPa·s

VHR(25° C.)=99.2%

30 VHR(80° C.)=98.0%

#### **EXAMPLE 5**

The composition comprising the following components was prepared.

Component I		
V-H1SiB(2F,3F)-O2	3.0%	
3-H1SiB(2F,3F)-O2	9.0%	
5-H1SiB(2F,3F)-O2	12.0%	4
3-HH1SiB(2F,3F)-1	5.0%	
2-HH1SiB(2F,3F)-O2	15.0%	
3-HH1SiB(2F,3F)-O2	10.0%	
3-HH1SiB(2F,3F)-O3	15.0%	
3-H2H1SiB(2F,3F)-O2	5.0%	
Component II		4
5-HHCF2OB(2F,3F)-O2	6.0%	
5-HBCF2OB(2F,3F)-O2	5.0%	
Component III		
3-HB-O2	4.0%	
3-HHB-O1	3.0%	5
1O1-HBBH-4	4.0%	
3-НВВН-3	4.0%	

The composition had the following physical properties:

Tc=65.5° C.

T<sub>1</sub><-20° C.

Δn=0.101

Δε=-3.5

η<sub>20</sub>=32.0 mPa·s

VHR(25° C.)=99.0%

VHR(80° C.)=98.4%

## **EXAMPLE 4**

The composition comprising the following components was prepared.

	Component I	,	
40	3-H1SiB(2F,3F)-O2 2-HH1SiB(2F,3F)-O2	6.0% 3.0%	
	3-HH1SiB(2F,3F)-O2 Component II	3.0%	
	3-DhB(2F,3F)-O2	7.0%	
	3-Dh2B(2F,3F)-O2	3.0%	
45	3-HDhB(2F,3F)-O2	10.0%	
	5-HDbB(2F,3F)-O2	10.0%	
	3-H2DhB(2F,3F)-O2	5.0%	
	3-BDhB(2F,3F)-O2	16.0%	
	5-BDhB(2F,3F)-O2	16.0%	
	3-DhHB(2F,3F)-O2	5.0%	
50	Component III		
	3-HH-4	3.0%	
	1O1-HH-3	3.0%	
	3-HB-O2	3.0%	
	3-HB-O4	4.0%	
e e	3-HEH-5	3.0%	

The composition had the following physical properties:

Tc=75.5° C.

 $T_L < -20^{\circ} \text{ C.}$ 

Δn=0.102

Δε=–4.7

η<sub>20</sub>=40.7 mPa·s

## **EXAMPLE 6**

Component I			Component I	
3-H1SiB(2F,3F)-O2	6.0%	5	3-H1SiB(2F,3F)-O2	5.0%
2-HH1SiB(2F,3F)-1	4.0%		V2-H1SiB(2F,3F)-O2	5.0%
3-HH1SiB(2F,3F)-O2	4.0%		2-HH1SiB(2F,3F)-O2	7.0%
Component II			3-HH1SiB(2F,3F)-V	3.0%
<del></del>			3-HH1SiB(2F,3F)-O2	12.0%
3-HCF2OB(2F,3F)-O2	3.0%		3-HH1SiB(2F,3F)-O3	7.0%
3-HOCF2B(2F,3F)-O2	3.0%	10	1V2-HH1SiB(2F,3F)-O2	5.0%
3-ChOCF2B(2F,3F)-O2	3.0%		Component II	
3-HHCF2OB(2F,3F)-O1	5.0%			
5-HHCF2OB(2F,3F)-O2	9.0%		3-DhB(2F,3F)-O2	7.0%
3-HBCF2OB(2F,3F)-O2	7.0%		3-HDhB(2F,3F)-O2	10.0%
5-HBCF2OB(2F,3F)-O2	7.0%		V2-HDhB(2F,3F)-O2	5.0%
3-HChOCF2B(2F,3F)-O1	14.0%	15	5-HDhB(2F3F)-O2	15.0%
3-HHOCF2B(2F,3F)-O1	4.0%	15	3-BDhB(2F,3F)-O2	4.0%
3-HBOCF2B(2F,3F)-O1	4.0%		5-BDhB(2F,3F)-O2	4.0%
3-HOCF2B(2F,3F)B(2F,3F)-O1	4.0%		Component III	
Component III				
			3-НН-4	3.0%
3-HH-4	5.0%	20	3-HB-O2	4.0%
3-HB-O2	6.0%	20	V-HHB-3	4.0%
3-HB-O4	4.0%			
3-НН-ЕМе	4.0%			
3-HHB-1	4.0%	Т	he composition had the following	ing physical r

The composition had the following physical properties: 25

Tc=80.1° C.

 $T_L < -20^{\circ} \text{ C.}$   $\Delta n = 0.094$ 

 $\Delta \epsilon = -3.1$ η<sub>20</sub>=36.1 mPa·s

**EXAMPLE 7** 

The composition comprising the following components was prepared.

Tc=72.8° C.

T<sub>L</sub><-20° C.

 $\Delta n = 0.104$  $\Delta \epsilon = -5.1$ 

η<sub>20</sub>=38.3 mPa·s VHR(25° C.)=99.2%

VHR(80° C.)=98.0%

#### **EXAMPLE 9**

The composition comprising the following components was prepared.

Component I		
3-H1SiB(2F,3F)-O2	5.0%	
2-HH1SiB(2F,3F)-O2	4.0%	4
Component II		•
3-DhB(2F,3F)-O2	5.0%	
3-HDhB(2F,3F)-O2	12.0%	
5-HDhB(2F,3F)-O2	8.0%	
3-BDhB(2F,3F)-O2	5.0%	
5-BDhB(2F,3F)-O2	5.0%	4
3-DhB(2F,3F)B(2F,3F)-O2	4.0%	
5-HHCF2OB(2F,3F)-Ó2	5.0%	
5-HBCF2OB(2F,3F)-O2	6.0%	
3-HCF2OHB(2F,3F)-O2	5.0%	
3-HChOCF2B(2F,3F)-O1	5.0%	
3-HCF2OBB(2F,3F)-O2	5.0%	5
3-HBOCF2B(2F,3F)-O1	5.0%	
3-HB(2F,3F)OCF2B(2F,3F)-O1	5.0%	
Component III		
3-НН-4	8.0%	
3-HB-O2	4.0%	5
3-HH-EMe	4.0%	-

The composition had the following physical properties:

Tc=94.0° C.

T<sub>L</sub><-20° C.

 $\Delta n = 0.099$ 

 $\Delta \epsilon = -4.5$ 

η<sub>20</sub>=42.0 mPa·s

#### **EXAMPLE 8**

The composition comprising the following components was prepared.

Component I  -H1SiB(2F,3F)-O2 -H1SiB(2F,3F)-O2 -HH1SiB(2F,3F)-O2 -HH1SiB(2F,3F)-O2 -HH1SiB(2F,3F)-O3 Component II	5.0% 5.0% 10.0% 12.0%
i-H1SiB(2F,3F)-O2 2-HH1SiB(2F,3F)-O2 3-HH1SiB(2F,3F)-O2 3-HH1SiB(2F,3F)-O3	5.0% 10.0% 12.0%
2-HH1SiB(2F,3F)-O2 3-HH1SiB(2F,3F)-O2 3-HH1SiB(2F,3F)-O3	10.0% 12.0%
-HH1SiB(2F,3F)-02 -HH1SiB(2F,3F)-03	12.0%
-HH1SiB(2F,3F)-O3	
-HH1SiB(2F,3F)-O3	12.0%
-HCF2OB(2F,3F)-O2	5.0%
-HHCF2OB(2F,3F)-O2	12.0%
5-HBCF2OB(2F,3F)-O2	8.0%
-HCF2OBB(2F,3F)-O2	4.0%
-HChOCF2B(2F,3F)-O1	4.0%
	4.0%
	8.0%
Component III	•
⁄-нн-3	3.0%
-HB-O2	4.0%
-НН-ЕМс	4.0%
3	-HHOCF2B(2F,3F)-O1 -HB(2F,3F)-OCF2B(2F,3F)-O2 -Component III /-HH-3 -HB-O2

The composition had the following physical properties:

Tc=75.3° C.

T<sub>L</sub><-20° C.  $\Delta n = 0.098$ 

 $\Delta \epsilon = -3.9$ 

η<sub>20</sub>=37.6 mPa·s

VHR(25° C.)=99.0% VHR(80° C.)=98.3%

## **EXAMPLE 10**

3-H1SiB(2F,3F)-O2	8.0%	5	3-H1SiB(2F,3F)-O2	8.0%
5-H1SiB(2F,3F)-O2	8.0%		5-H1SiB(2F,3F)-O2	8.0%
2-HH1SiB(2F,3F)-O2	10.0%		2-HH1SiB(2F,3F)-O2	10.0%
3-HH1SiB(2F,3F)-O2	12.0%		3-HH1SiB(2F,3F)-O2	12.0%
3-HH1SiB(2F,3F)-O3	10.0%		3-HH1SiB(2F,3F)-O3	6.0%
Component II	201070		3-HB1SiB(2F,3F)-O2	4.0%
<u>compositor is</u>		10	Component II	
3-DhB(2F,3F)-O2	5.0%			
5-DhB(2F,3F)-O2	5.0%		3-HDhB(2F,3F)-O2	3.0%
Component III			3-BDhB(2F,3F)-O2	3.0%
			5-HHCF2OB(2F,3F)-O2	3.0%
3-HH-4	4.0%		5-HBCF2OB(2F,3F)-O2	3.0%
3-HEH-5	4.0%	15	Component III	
3-HB-O2	8.0%	13	<del></del>	
3-HEB-O3	4.0%		3-HB-O2	10.0%
4-HEB-O1	4.0%		3-HH-EMe	4.0%
1O1-HBBH-4	6.0%		3-HEB-O3	4.0%
1O1-HBBH-5	6.0%		4-HEB-O1	4.0%
3-НВВН-3	3.0%	20	3-HHB-3	4.0%
3-HBB(F)H-3	3.0%	20	101-HBBH-4	7.0%
		<del></del>	The other component	
manition had the following	ina abraical arcacet	ian.	3-ННЕВН-3	4.0%
position had the follow	mg buysicai broben	102:	3-ННЕВН-5	3.0%

T<sub>L</sub><-20° C.

Δn=0.098

Δε=-2.9

 $\eta_{20}$ =30.0 mPa·s

**EXAMPLE 11** 

The composition comprising the following components was prepared.

The composition had the following physical properties:

Tc=80.1° C. T<sub>L</sub><-20° C.

 $\Delta n = 0.098$ 

Δε≕-2.6 η<sub>20</sub>=30.1 mPa·s

**EXAMPLE 13** 

The composition comprising the following components was prepared

Component I			Component I	
3-H1SiB(2F,3F)-O2	5.0%		3-H1SiB(2F,3F)-O2	5.0%
5-H1SiB(2F,3F)-O2	5.0%	40	3-HH1SiB(2F,3F)-O2	5.0%
2-HH1SiB(2F,3F)-O2	15.0%		Component II	
3-HH1SiB(2F,3F)-O2	15.0%		<u> </u>	
3-HH1SiB(2F,3F)-O3	14.0%		3-DhB(2F,3F)-O2	7.0%
3-H2H1SiB(2F,3F)-O2	5.0%		3-HDhB(2F,3F)-O2	15.0%
3-H1SiHB(2F,3F)-O2	5.0%		5-HDbB(2F,3F)-O2	15.0%
Component II		45	3-BDhB(2F,3F)-O2	10.0%
			Component III	
3-HHCF2OB(2F,3F)-1	4.0%			
3-HHCF2OB(2F,3F)-O2	6.0%		2-HH-3	5.0%
5-HHCF2OB(2F,3F)-O2	5.0%		3-HH-4	5.0%
Component III			3-HB-O2	4.0%
		50	3-HB-O4	5.0%
3-HB-O2	10.0%		3-HEH-3	4.0%
3-HB-O4	4.0%		3-HH-EMe	4.0%
3-HEH-3	4.0%		3-HBBH-3	4.0%
3-HHB-1	3.0%		3-HBB(F)H-5	4.0%
			The other component	
		55		
mposition had the follow	ing physical proper	ties:	3-ННЕН-3	8.0%

The composition had the following physical properties:

Tc=68.7° C.

T<sub>L</sub><-20° C.

 $\Delta n = 0.094$ 

Δε=-3.6

η<sub>20</sub>=26.0 mPa·s

#### **EXAMPLE 12**

The composition comprising the following components was prepared.

The composition had the following physical properties:

Tc=91.5° C.

T<sub>r</sub><-20° C.

 $\Delta n = 0.089$ 

Δε≕-3.5

η<sub>20</sub>=32.7 mPa·s

## **EXAMPLE 14**

			<u> </u>	
Component I			Component I	
3-H1SiB(2F,3F)-O2	6.0%	5	3-H1SiB(2F,3F)-O2	5.0%
2-HH1SiB(2F,3F)-O2	6.0%		5-H1SiB(2F,3F)-O2	6.0%
Component II			2-HH1SiD(2F,3F)-O2	6.0%
-			3-HH1SiB(2F,3F)-O2	9.0%
3-HCF2OB(2F,3F)-O2	5.0%		3-HB1SiB(2F,3F)-O2	3.0%
3-HOCF2B(2F,3F)-O2	5.0%		3-H1SiHB(2F,3F)-O2	4.0%
5-HHCF2OB(2F,3F)-O2	10.0%	10	Component II	
3-HCF2OHB(2F,3F)-02	5.0%			
5-HBCF2OB(2F,3F)-O2	15.0%		3-DhB(2F,3F)-O2	5.0%
5-HChOCF2E(2F,3F)-O1	5.0%		3-HDhB(2F,3F)-O2	5.0%
3-HBOCF2B(2F,3F)-O1	5.0%		V-HDhB(2F,3F)-O2	5.0%
Component III			5-HDhB(2F,3F)-O2	11.0%
		15	3-BDhB(2F,3F)-O2	5.0%
3-HH-4	12.0%	13	Component III	
3-HB-O2	12.0%			
3-НН-ЕМе	6.0%		2-HH-3	5.0%
3-HHB-3	8.0%		V-HH-5	10.0%
		-	3-HB-O2	8.0%
		20	3-HB-O4	4.0%
The composition had the following	The composition had the following physical properties:		V-HHB-3	3.0%
The composition had the following physical properties:			1O1-HBBH-5	6.0%
Tc=76.8° C.				
T <sub>L</sub> <-20° C.			The composition had the following	ing physical properties:
Δn=0.086		25	Tc=71.5° C.	
ΔΠ=0.080		4.7	T <sub>1</sub> <-20° C.	
$\Delta \epsilon = -2.6$ $\eta_{20}32\ 26.8\ \text{mPa·s}$			Δn=0.095	
			$\Delta \epsilon = -3.5$	
			η <sub>20</sub> =27.5 mPa·s	
EXAMPLE 15		30	<del></del>	17
			EXAMPLE :	
The composition comprising the fe	ollowing components	•	The composition comprising the	following components

was prepared.

was prepared.

Component I			Component I	
2-HH1SiB(2F,3F)-O2	5.0%		3-H1SiB(2F,3F)-O2	5
3-HH1SiB(2F,3F)-O2	6.0%		5-H1SiB(2F,3F)-O2	5
Component II		40	2-HH1SiB(2F,3F)-O2	10
			3-HH1SiB(2F,3F)-O2	12
3-HDhB(2F,3F)-O2	6.0%		Component II	
5-HDhB(2F,3F)-O2	12.0%			
3-DhH2B(2F,3F)-O2	6.0%		3-HCF2OB(2F,3F)-O2	8
5-HHCF2OB(2F,3F)-O2	8.0%		3-HOCF2B(2F,3F)-O2	8
5-HBCF2OB(2F,3F)-O2	8.0%	45	5-HHCF2OB(2F,3F)-O2	4
3-HChOCF2B(2F,3F)-O1	5.0%	43	3-HChOCF2B(2F,3F)-O1	5
3-HDOCF2B(2F,3F)-O1	5.0%		3-HB(2F,3F)OCF2B(2F,3F)-O1	3
Component III			Component III	
3-НН-4	12.0%		2-НН-3	4
V2-HH-4	5.0%		3-HH-4	9
3-HB-O2	12.0%	50	3-HB-O2	8
2-HH-EMe	4.0%		3-HB-O4	-
3-HH-EMe	6.0%		3-HHB-3	
			1O1-HBBH-5	

The composition had the following physical properties: 55

Tc=87.5° C.

T<sub>L</sub><-20° C.

 $\Delta n = 0.087$ 

Δε=-3.5

 $\eta_{20}$ =29.5 mPa·s

## **EXAMPLE 16**

The composition comprising the following components was prepared.

The composition had the following physical properties:

5.0%

To=65.2° C. T<sub>L</sub><-20° C.

3-ННЕН-3

Δn=0.084

Δε=−2.7

η<sub>20</sub>=24.6 mPa·s

## **EXAMPLE 18**

Component I			Component I	
3-H1SiB(2F,3F)-O2	5.0%	5	3-H1SiB(2F,3F)-O2	7.0%
5-H1SiB(2F,3F)-O2	5.0%		3-HH1SiB(2F,3F)-O2	7.0%
2-HH1SiB(2F,3F)-O2	10.0%		Component II	
3-HH1SiB(2F,3F)-O2	12.0%			
Component II			5-HHCF2OB(2F,3F)-O2	6.0%
			5-HBCF2OB(2F,3F)-O2	4.0%
3-HDhB(2F,3F)-O2	8.0%	10 .	5-HChOCF2B(2F,3F)-O1	5.0%
5-HDhB(2F,3F)-O2	8.0%		Component III	
3-HOCF2B(2F,3F)-O2	8.0%			
5-HHCF2OB(2F,3F)-O2	3.0%		3-HH-4	12.0%
5-HBCF2OB(2F,3F)-O2	5.0%		3-HB-O2	16.0%
Component III			3-HB-O4	4.0%
		15	2-HH-EMe	5.0%
2-HH-3	5.0%	15	3-НП-ЕМе	16.0%
3-HH-4	8.0%		3-HHB-1	8.0%
3-HB-O2	8.0%		3-HHB-3	10.0%
3-HEB-O3	4.0%			
4-HEB-O1	4.0%			
3-HHB-3	3.0%	20		
1O1-HBBH-5	4.0%	20	The composition had the following	ig physical p

The composition had the following physical properties:

Tc=68.1° C.

T<sub>L</sub><-20° C.

 $\Delta n = 0.086$ 

 $\Delta \epsilon = -3.2$ 

 $\eta_{20}$ =26.8 mPa·s

#### **EXAMPLE 19**

The composition comprising the following components was prepared.

Tc=71.6° C.

T<sub>L</sub><-20° C.

Δn=0.077 25

30

35

 $\Delta \epsilon = -1.1$ 

η<sub>20</sub>=14.4 mPa·s

## **EXAMPLE 21**

The composition comprising the following components was prepared.

Component I	
2-HH1SiB(2F,3F)-O2	6.0%
3-HH1SiB(2F,3F)-O2	6.0%
Component II	
3-HDhB(2F,3F)-O2	6.0%
5-HDhB(2F,3F)-O2	6.0%
Component III	
2-НН-3	6.0%
3-HH-4	10.0%
3-HB-O2	16.0%
3-HB-O4	4.0%
2-НН-ЕМе	4.0%
3-НН-ЕМс	15.0%
3-HHB-1	8.0%
3-HHB-3	8.0%
3-HHB-O1	5.0%

The composition had the following physical properties:

Tc=80.4° C.

T<sub>L</sub><-20° C.

Δn=0.077

Δε=-1.4

η<sub>20</sub>=14.3 mPa·s

#### **EXAMPLE 20**

The composition comprising the following components was prepared.

	Component I	
	3-H1SiB(2F,3F)-O2	7.0%
	3-HH1SiB(2F,3F)-O2	5.0%
)	3-HH1SiB(2F,3F)-O3	5.0%
	Component II	
	5-HDhB(2F,3F)-O3	6.0%
	3-HCF2OB(2F,3F)-O2	5.0%
	5-HBCF2OB(2F,3F)-O2	5.0%
5	Component III	
	3-HH-4	12.0%
	3-HB-O2	16.0%
	2-НН-ЕМе	5.0%
	3-НН-ЕМе	16.0%
0	3-HHB-1	8.0%
1	3-HHB-3	10.0%

The composition had the following physical properties:

Tc=68.3° C.

T<sub>L</sub><-20° C.

Δn=0.076

Δε=-1.4

 $\eta_{20}$  32 15.3 mPa·s

## **EXAMPLE 22**

Component I			Component I	
2-HH1SiB(2F,3F)-O2	10.0%	5	3-H1SiB(2F,3F)-O2	5.0%
3-HH1SiB(2F,3F)-O2	7.0%		5-H1SiB(2F,3F)-O2	5.0%
3-HB1SiB(2F,3F)-O2	3.0%		2-HH1SiB(2F,3F)-O2	10.0%
Component II	•		3-HH1SiB(2F,3F)-O2	10.0%
-			Component II	
3-DhB(2F,3F)-O2	5.0%			
3-HDhB(2F,3F)-O2	10.0%	10	3-HDhB(2F,3F)-O2	5.0%
5-HDhB(2F,3F)-O2	10.0%		5-HDbB(2F,3F)-O2	5.0%
3-BDhB(2F,3F)-O2	5.0%		3-HHCF2OB(2F,3F)-O2	5.0%
Component III			3-HBCF2OB(2F,3F)-O2	5.0%
			Component III	
3-HH-4	8.0%			
3-HB-O2	8.0%	15	2-HH-5	4.0%
3-HH-EMc	5.0%		3-HH-4	4.0%
3-HHB-3	4.0%		3-HB-O2	4.0%
3-НВВН-3	5.0%		3-HB-O4	4.0%
Component IV			3-HHB-3	4.0%
<del></del>			Component IV	
3-HB(2F,3F)-O2	5.0%	20		
5-HB(2F,3F)-O2	5.0%	20	3-HB(2F,3F)-O2	5.0%
3-HBB(2F,3F)-O2	5.0%		V-HB(2F,3F)-O2	5.0%
3-HHB(2F,3F)-O2	5.0%		3-HBB(2F,3F)-O2	5.0%
		_	V-HBB(2F,3F)-O2	5.0%
			3-HHB(2F,3F)-O2	5.0%
composition had the follow	ving physical properties	:	5-HHB(2F,3F)-O2	5.0%

TC=90.3° C.

 $T_L < -20^{\circ} \text{ C}.$ 

 $\Delta \hat{n} = 0.100$ 

**Δ∈=-4.1** 

η<sub>20</sub>=33.3 mPa·s

#### **EXAMPLE 23**

The composition comprising the following components was prepared.

Component I		
3-H1SiB(2F,3F)-O2	6.0%	
2-HH1SiB(2F,3F)-O2	6.0%	
3-HH1SiB(2F,3F)-O2	6.0%	
Component II		
3-HOCF2B(2F,3F)-O2	10.0%	
5-HHCF2OB(2F,3F)-O2	10.0%	
3-HChOCF2B(2F,3F)-O1	5.0%	
Component III		
3-НН-4	8.0%	
3-НН-ЕМе	5.0%	
3-HHB-3	4.0%	
Component IV		
3-HB(2F,3F)-O2	4.0%	
3-H2B(2F,3F)-O2	4.0%	
5-HB(2F,3F)-O2	8.0%	
5-HBB(2F,3F)-3	4.0%	
3-HBB(2F,3F)-O2	6.0%	
3-H2BB(2F,3F)-O2	4.0%	
3-HHB(2F,3F)-O2	6.0%	
V-HHB(2F,3F)-O2	4.0%	

The composition had the following physical properties:

Tc=78.1° C.

T<sub>L</sub><-20° C.  $\Delta n = 0.100$ 

 $\Delta \epsilon = -3.8$ 

η<sub>20</sub>=31.0 mPa·s

## **EXAMPLE 24**

The composition comprising the following components was prepared.

The composition had the following physical properties:

Tc=79.0° C.

T<sub>L</sub><-20° C.

 $\Delta n = 0.103$ 

Δε=-4.1

35

 $\eta_{20}$ =28.5 mPa·s

VHR(25° C.)=99.1%

VHR(80° C.)=97.9%

## **EXAMPLE 25**

The composition comprising the following components was prepared.

Component I	
3-H1SiB(2F,3F)-O2	9.0%
5-H1SiB(2F,3F)-O2	8.0%
3-HH1SiB(2F,3F)-O2	12.0%
5-HH1SiB(2F,3F)-O2	6.0%
3-HH1SiB(2F,3F)-O3	12.0%
Component II	
3-HBOCF2B(2F,3F)-O2	16.0%
5-HBOCF2B(2F,3F)-O2	16.0%
Component III	
3-НН-4	11.0%
3-HB-O2	10.0%

The composition had the following physical properties:

Tc=62.3° C. T<sub>L</sub><-20° C.

 $\Delta n = 0.095$ 

Δε=-3.7

η<sub>20</sub>=28.6 mPa·s

VHR(25° C.)=98.8% VHR(80° C.)=97.8%

## **EXAMPLE 26**

-conf	

(III-2)

Component I	
3-H1SiB(2F,3F)-O2 5-H1SiB(2F,3F)-O2 3-HH3SiB(2F,3F)-O2	10.0% 9.0% 12.0%
5-HH1SiB(2F,3F)-O2 3-HH1SiB(2F,3F)-O3 Component II	5.0% 12.0%
3-HHCF2OB(2F,3F)-O2 5-HHCF2OB(2F,3F)-O2 Component III	14.0% 14.0%
3-HII-4 3-HB-O2	11.0% 13.0%

The composition had the following physical properties:

Tc=69.4° C.

T, <-20° C.

 $\Delta n = 0.085$ 

 $\Delta \epsilon = -3.4$ 

η<sub>20</sub>=26.5 mPa·s

VHR(25° C.)=99.0%

VHR(80° C.)=98.2%

#### INDUSTRIAL APPLICABILITY

As shown in the Examples, the present invention provides a liquid crystal composition which has a suitable An in accordance with cell thickness, in particular a largely negative dielectric anisotropy, a broad nematic liquid crystal phase range, a high voltage holding ratio and a low viscosity while satisfying various properties required for liquid crystal compositions for AM-LCD.

What is claimed is:

1. A liquid crystal composition comprising Component I selected from the group consisting of the compounds represented by formula (I), Component II selected from the group consisting of the compounds represented by formula (II) and Component III selected from the group consisting of the compounds represented by formulas (III-1) and (III-2)

$$R^{1} \longrightarrow (Z^{1} - A^{1})_{\overline{m}} \longrightarrow Z^{2} \longrightarrow R^{2}$$

$$R^{1} \longrightarrow (A^{2} - Z^{3})_{\overline{n}} \longrightarrow A^{3} \longrightarrow Z^{4} \longrightarrow R^{2}$$
(III)

$$R^{1}$$
  $(COO)_{\overline{p}}$   $\Lambda^{4}$   $(COO)_{\overline{q}}$   $R^{3}$ 

$$R^{1}$$
  $A^{5}$   $R^{3}$ 

wherein each R1 independently represents an alkyl group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms; each R<sup>2</sup> independently represents an alkyl or alkoxy group having 1-10 carbon atoms, or an alkenyl group having 2-10 carbon atoms; Z<sup>1</sup> and Z<sup>2</sup> represent -CH<sub>2</sub>SiH<sub>2</sub>-, a single bond or -CH<sub>2</sub>CH<sub>2</sub>-, with the proviso that one of Z<sup>I</sup> and Z<sup>2</sup> represents -CH<sub>2</sub>SiH<sub>2</sub>- and the other represents a single bond or -CH<sub>2</sub>CH<sub>2</sub>-; ring A<sup>1</sup> represents a trans-1,4-cyclohexylene group, or a 1,4phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; m is 0 or 1, with the proviso that when m is 0, Z<sup>2</sup> represents —CH<sub>2</sub>SiH<sub>2</sub>—; Z<sup>3</sup> and Z<sup>4</sup> each independently represent a single bond,  $-CH_2CH_2$ —,  $-CF_2O$ — or  $-OCF_2$ —; rings  $A^2$  and  $A^3$ represent a tetrahydropyran-2,5-diyl group, a trans-1,4cyclohexylene group, a cyclohexa-1-ene-1,4-diyl group, or a 1,4-phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; n is 0 or 1; with the proviso that when n=0 and Z4 represents a single bond or -CH<sub>2</sub>CH<sub>2</sub>—, then ring A<sup>3</sup> represents a tetrahydropyran-2, 5-diyl group; when n=0 and Z4 represents -CF2O or -OCF<sub>2</sub>—, then ring A<sup>3</sup> represents a trans-1,4cyclohexylene group or a cyclohexa-1-ene-1,4-diyl group; when n=1 and  $Z^3$  and  $Z^4$  each independently represent a single bond or -CH2CH2-, then one of rings A2 and A3 represents a tetrahydropyran-2,5-diyl group; when n=1 and and/or Z<sup>4</sup> represent —CF<sub>2</sub>O— or —OCF<sub>2</sub>—, then rings and A<sup>3</sup> represent a trans-1,4-cyclohexylene group, a cyclohexa-1-ene-1,4-diyl group, or a 1,4-phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; each R3 independently represents an alkyl group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms, in which one -CH2- may be substituted with -O-; ring A<sup>4</sup> represents a 1,4-phenylene group or a trans-1,4-cyclohexylene group; ring A<sup>5</sup> represents a trans-1,4-cyclohexylene group, or a 1,4-phenylene group in which one or more hydrogen atoms may be substituted with fluorine atoms; p, q and s are each independently 0 or 1 with the proviso that (p+q) is 0 or 1.

A liquid crystal composition as claimed in claim 1, which comprises 3-80% by weight of Component I, 3-80% by weight of Component II and 1-80% by weight of Component III.

3. A liquid crystal composition as claimed in claim 1, which further comprises Component IV selected from the group consisting of the compounds represented by formula (IV)

$$R^4$$
— $(Z^5$ — $A^6)_1$ — $Z^6$ — $R^5$ 

wherein R<sup>4</sup> represents an alkyl group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms, R<sup>5</sup> represents an alkyl or alkoxy group having 1-10 carbon atoms or an alkenyl group having 2-10 carbon atoms, Z<sup>5</sup> and

Z<sup>6</sup> each independently represent a single bond or —CH<sub>2</sub>CH<sub>2</sub>—, ring A<sup>6</sup> represents a 1,4-phenylene group or a trans-1,4-cyclohexylene group, and t is 0 or 1.

4. A liquid crystal composition as claimed in claim 1, wherein the clearing point (Tc) is 60 to 100° C., the

refractive anisotropy (An) is 0.06 to 0.12 and the dielectric anisotropy (Δε) is -6 to -1.
5. A liquid crystal display element containing the liquid

crystal composition as claimed in claim 1.

## UNITED STATES PATENT AND TRADEMARK OFFICE **CERTIFICATE OF CORRECTION**

PATENT NO. : 6,395,353 B2 DATED

: May 28, 2002

INVENTOR(S): Motoki Yanai et al.

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 23,

Line 28, change " $\eta_{20}$ 32 26.8 mPa•s" to --  $\eta_{20}$  = 26.8 mPa•s --; Line 47, change "3-HDOCF2B(2F,3F)-O1" to -- 3-HBOCF2B(2F,3F)-O1 --.

Column 24,

Line 7, change "2-HH1SiD(2F,3F)-O2" to -- 2-HH1SiB(2F,3F)-O2 --.

Column 29,

Line 36, change "An" to  $-\Delta n$  --.

Signed and Sealed this

Fifth Day of November, 2002

Attest:

JAMES E. ROGAN Director of the United States Patent and Trademark Office

Attesting Officer